# DESIGN, DEVELOPMENT AND PERFORMANCE EVALUATION OF A MODIFIED UASB REACTOR FOR TREATMENT OF LOW-STRENGTH WASTEWATERS

*A Thesis Submitted*
*in Partial Fulfilment of the Requirements*
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*by*

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*to the*

**DEPARTMENT OF CIVIL ENGINEERING**
INDIAN INSTITUTE OF TECHNOLOGY KANPUR

June, 1996

Dedicated to

*My Fellow Citizens*

for the Cause of a Better Environment

# CERTIFICATE

It is certified that the work contained in the thesis entitled *"Design, Development and Performance Evaluation of a Modified UASB Reactor for Treatment of Low-strength Wastewaters"*, by **Grasius M.G.**, has been carried out under my supervision and that this work has not been submitted elsewhere for a degree.

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| | |
|---|---|
| $c_1$ and $c_2$ | Constants, characteristic of the different anaerobic treatment process |
| $d$ | Diameter of the tube (L) |
| $E$ | Efficiency of organic matter removal (%) |
| $F$ | Degradable particulate organic matter ($ML^{-3}$) |
| $F_o$ | Initial concentration of degradable particulate organic matter ($ML^{-3}$) |
| $k$ | Maximum specific substrate utilisation rate ($T^{-1}$) |
| $K_d$ | Microorganism decay coefficient ($T^{-1}$) |
| $k_h$ | Hydrolysis rate constant ($T^{-1}$) |
| $K_s$ | Half-saturation constant ($ML^{-3}$) |
| $l$ | Length of the tube (L) |
| $L_t$ | Relative length of the tube |
| $L'_t$ | Relative length for transition region |
| $q$ | Specific substrate utilisation rate ($T^{-1}$) |
| $S$ | Residual growth limiting substrate concentration ($ML^{-3}$) |
| $V_o$ | The average flow velocity through the tube ($LT^{-1}$) |
| $V_{sc}$ | Critical settling velocity ($LT^{-1}$) |
| $X$ | Concentration of biomass ($ML^{-3}$) |

| | |
|---|---|
| $Y$ | Yield coefficient |
| $\mu$ | Specific growth rate of biomass ($T^{-1}$) |
| $\mu_m$ | Maximum specific growth rate ($T^{-1}$) |
| $\nu$ | Kinematic viscosity of the wastewater ($L^2T^{-1}$) |
| $\theta$ | Angle of inclination of the tube with horizontal |
| $\theta_h$ | Hydraulic retention time (T) |

# SYNOPSIS

With the advent of several high-rate treatment systems, anaerobic process has emerged as a promising alternative for the treatment of many types of wastewaters. Retention of active biomass independent of hydraulic retention time (HRT), better contact between the biomass and the waste organic matter and higher waste stabilisation capacity per unit weight of the microbial film/granules that develop in the reactor are the reasons for the improved process efficiency and stability at relatively low HRTs in these high-rate reactors. Upflow Anaerobic Sludge Blanket (UASB) reactors are the most popular among these "retained biomass" systems. Recent experience with full scale UASB reactors treating domestic wastewater has demonstrated the suitability of the process in municipal waste management in tropical regions. However, this treatment system still suffers from certain limitations challenging its effectiveness especially for the treatment of low strength wastewaters.

Microbial granulation is difficult in UASB reactors with low strength wastewaters, as achievement of the required loading rate is restricted by the maximum superficial velocity that can retain the flocculent seed sludge. For various reasons, presence of granular sludge in the reactor is advantageous for the success of UASB reactors. The improved specific waste stabilisation capacity of granular sludge due to the syntrophic association of the different groups of anaerobic microbial consortium is one among them. Another limitation of the conventional UASB reactor is the possibility of impaired bed expansion

and microbial contact with the substrate due to the low gas production rate during treatment of low strength wastewaters especially at low temperatures. Literature on the UASB full-scale experience indicates that a considerable fraction of the effluent COD is constituted by suspended organic matter.

Thus, there is a need to develop a reactor configuration with a more effective gas-liquid-solid separation (GLSS) device which can minimise the escape of suspended organic matter from the effluent at extremely low HRTs. This would result in the production of effluents with low COD. Simultaneously, the reactor should be effective in the retention of flocculent seed sludge at low HRTs demanded by the loading rates favourable for granulation.

With this aim, a reactor configuration was conceived and designed by incorporating tube settlers in the settler zone replacing the conventional GLSS of a UASB reactor. An assembly of PVC tubes, of required diameter and length may be kept inclined over the sludge zone. These tubes can be supported on polyethylene mesh of proper opening size to permit passage of sludge. This arrangement of the tubes is expected to effect proper gas-liquid-solid separation. As the biomass with the adhering gas bubble moves along the top inside surface of the tubes, the frictional resistance offered by the tube surface to the gas bubble is expected to effect the separation of gas bubbles from the biomass.

Two units of the modified UASB reactors, $R_1$ and $R_2$ with the settlers inclined at angle of $45^o$ and $60^o$ were used in this study. These settlers contained 19 PVC tubes of 2 cm diameter and 54 cm length. Both the reactors had an empty bed liquid volume of 9.16 litres each and

an axial liquid length of 1.24 m. The sludge zone height and the settler zone length of the reactors were 57 cm each. These reactors were used to develop granular sludge from flocculent type seed sludge using low strength synthetic sucrose wastewater. Performance of the reactor at HRTs ranging from 5-1 h were evaluated using wastewater containing sucrose as well as complex organics. Thereafter, these reactors with the granular sludge were used to treat raw domestic wastewater of low strength. All through this study the reactors were operated at ambient temperature. A brief discussion of the results obtained in these studies is presented below.

During the first phase of the study the reactors were seeded with flocculent type sludge taken from the 5 MLD UASB plant treating domestic wastewater of Kanpur (India). Both the reactors $R_1$ and $R_2$ were fed with sucrose based synthetic wastewater of 485 mg/L COD. The "primary start-up" was over in about 50 days of continuous operation. The reactor VSS increased to 8.14 g/L and 7.53 g/L in $R_1$ and $R_2$ respectively from the initial value of 6 g/L. The soluble COD removed efficiency in the reactors were above 80% at a loading rate of 2.33 g COD/L.d corresponding to an HRT of 5 h. The specific substrate utilisation rate was 0.24 g COD/g VSS.d. These reactors were further operated for about 170 days with the same wastewater to evaluate the pseudo steady state (PSS) performance at 5, 4, 3, 2.4 and 2 h HRT. By day 72 the sludge level in the reactor had reached to a height of 50 cm. Thereafter, excess sludge was withdrawn from port No. 2 provided at a height of 50 cm so as to prevent the entry of sludge bed to the settler zone.

Presence of a large number of granules was observed in the reactor sludge taken out during the PSS operation at 4 h HRT. The corresponding space and sludge loading rates were 2.88 g COD/L.d and 0.24 g COD/g VSS.d respectively. These values are relatively low compared to those reported loading rates that are conducive for granulation. The observed gas production rate in the reactor was about 0.9 L/L.d. It was concluded that apart from the loading rate, the gas production rate which influences the bed agitation greatly, is also a factor that contributes to granulation.

Reactor performance data collected during PSS at HRTs ranging from 2-5 h show that the total COD removal efficiency was in the range of 83-90% in both the reactors. The soluble COD removal efficiencies exhibited by the reactors varied from 91-95%. The total $BOD_5$ of the effluent ranged from 17-27 mg/L at HRTs ranging from 5-2.4 h. Thus the tube settlers were found to be very effective in reducing the suspended fraction of the effluent COD.

Maximum reactor VSS of 13.38 g/L was observed at 3 h HRT. The specific substrate utilisation rate increased from 0.21-0.52 g COD/g VSS.d as the HRT decreased from 5 to 2 h. This indicated improved microbial activity at higher loads. The VSS/TSS ratio of the sludge bed increased with a decrease in HRT. This was due to the continuous enrichment of the reactor sludge with the newly synthesised biomass from sucrose wastewater. The average specific methanogenic activity (SMA) of the reactor sludge was about 1.0 g $CH_4$-COD/g VSS.d. Methane recovered from the reactor during this phase of the study was 413-1076 mL/L.d. The methane production rate (including that lost with effluent) was

0.13-0.37 g $CH_4$ COD/g VSS.d. The average methane and sludge yield at these HRTs were respectively 0.7 g $CH_4$-COD/g COD removed and 0.16 g VSS/g COD removed. The regular sludge wasting from port No. 2 has contributed significantly to the relatively low BSRTs and the resulting high sludge yield.

In the second phase of the study, these reactors with the granular sludge developed during the earlier phase of the study were used to treat complex synthetic wastewater prepared from a commercial grade baby food containing insoluble carbohydrate, protein and fat (CERELAC, NESTLE INDIA LTD.). Initially, the granular sludge was adapted to this wastewater during a "secondary start-up" operation at 4 h HRT. Thereafter the reactor $R_1$ was operated at HRTs of 4 h and 2 h and $R_2$ was operated at 4, 2 and 1 h to get PSS conditions. The total and soluble COD of the wastewater were 476 and 205 mg/L respectively. This phase of the study lasted for about 115 days.

After 45 days of initial reactor operation at 4 h HRT the total and soluble COD removal observed were 83% and 80% respectively. The reactor pH could be maintained above 7.1 without the addition of bicarbonate. The reactor temperature during this period was around 15$^{o}$C. These observations indicated a good adaptation of the granular sludge to the new wastewater.

During steady state operation at 4 h and 2 h HRT, the total COD removal efficiency was in the range of 81-83%. The soluble COD removal in settler zone was only marginal. The total $BOD_5$ of the effluent from the reactors was in the range of 28-35 mg/L at these HRTs. These results indicated that settlers were effective in retaining the

suspended solids while treating the complex wastewater. However, at 1 h HRT the tube settlers were not effective in achieving proper gas-liquid-solid separation.

The specific substrate utilization rate and VSS/TSS ratio increased with the loading rate. The SMA of the reactor sludge at 4 h and 2 h HRT were respectively 0.83 and 1.6 g $CH_4$-COD/g VSS.d. The volume of methane that could be recovered at these HRTs were 336 and 608 mL/L.d. The corresponding methane production rates were calculated to be 0.17 and 0.38 g $CH_4$-COD/g VSS.d. The average methane and sludge yield observed were respectively 0.47 g $CH_4$-COD/g COD removed and 0.16 g VSS/g COD removed. During this phase of the study also regular sludge wasting from port No. 2 contributed significantly to the reduction of BSRT in the reactor leading to high sludge yield.

During the final phase of the study the reactors were fed with domestic wastewater collected from a sump well in the residential area of I.I.T. Kanpur (India). The total COD of the wastewater varied between 125 and 622 mg/L. The reactors were operated at 3, 2 and 1 h HRT. After collecting PSS data at these HRTs, reactors $R_1$ and $R_2$ were operated without the tubes in the settler zone at 1 h and 2 h HRT respectively. This phase of the study lasted for about 125 days.

The granular sludge grown on CERELAC wastewater could be adapted easily to the domestic wastewater. Within 23 days of reactor operation at 3 h HRT, the maximum total and soluble COD removal efficiency in $R_1$ reached 86% and 74% respectively. In the case of $R_2$ which was operating at 2 h HRT, the maximum total and soluble COD removal efficiencies were 84% and 66% respectively.

Total COD removal efficiency at various HRTs employed was in the range of 78-87%. Corresponding to 1 h HRT, the space loading in the reactor was 9.65 g COD/L.d and at this condition the COD removal efficiency was 87%. The average total $BOD_5$ of the effluent was around 15 mg/L at all these HRTs. When the reactors were operated without tubes at 2 h and 1 h HRT, the COD removal efficiency decreased to 69% and 36%. This indicated the usefulness of providing tube settler in reducing the effluent COD. It was also observed that the reactor configuration was effective in keeping the effluent quality fairly stable in spite of the wide fluctuations in the influent COD and TSS concentrations.

Over the range of HRTs studied the SMA was in the range of 0.2 to 0.12 g $CH_4$-COD/g VSS.d. No gas could be recovered from the domestic wastewater indicating that the gas produced was less than that would escape with effluent in the dissolved form. Moreover, it was concluded that at the relatively low HRTs, the liquefaction and biomethanation of the trapped suspended organic matter was impaired. The average excess VSS production with respect to influent COD was 0.14 g COD/g $COD_{in}$. The average excess TSS production in the reactor was only $7.6 \times 10^{-2}$ g TSS/g $TSS_{in}$ which indicated accumulation of inorganic compounds in the reactor.

The sludge taken out from the reactor at 2 h and 1 h HRT were subjected to settling analysis. About 28% of the sludge particles were having settling velocities more than 50 m/h, which is the reported typical settling velocity of granules.

Studies were carried out to compare the performance of the sludge bed as well as the settler while the reactors were treating different types of wastewaters. The sludge drawn from different heights of the sludge bed were used to study the performance of the sludge bed.

In the case of wastewater containing insoluble organic matter, hydrolysis and acid formation were found to be prominent over a higher percentage of the sludge bed height compared to soluble wastewater. Methanogenesis was prominent over the top one third height of the sludge zone. The VSS concentration and VSS/TSS ratio of the reactor sludge was greatly dependent on type of wastewater. In the case of sucrose or CERELAC wastewater the above values were relatively high. This was attributed to the low inorganic content in the influent, accumulation of newly synthesised sludge and the effect of agitation due to the improved gas production which would lead to the formation of thick sludge bed. During domestic wastewater feeding the reactor VSS concentration and VSS/TSS ratio were relatively low in the reactors.

The SMA of the sludge from the reactor taken during the treatment of different types of wastewater indicated that the activity of the sludge was relatively high when the reactors were on sucrose and CERELAC based wastewater compared to that during domestic wastewater feeding. This indicated the accumulation of non-microbial organic matter in the sludge bed.

The advantage of tube settler in the settler zone was evident while treating all the three types of wastewaters. The soluble COD removal in this zone was marginal. The settlers were very effective in removing the suspended fraction of COD. A comparison of COD removal efficiency

based on total influent and filtered effluent-COD (TI-FE)-with total COD removal efficiency has shown that the reactors were 90-96% close to an ideal system, over the range of HRTs studied with the three types of wastewater except at 1 h HRT with CERELAC wastewater. In the later case this closeness was only 71%.

Close observation during reactor operation revealed that sludge particles/flocs ejected from the sludge bed with the gas were effectively settled in the tubes. Sludge particles attached with gas bubbles were also getting settled after releasing the gas bubbles. An examination of the tubes taken out of the reactor after completion of the study showed that the settled suspended solids were sliding back to the sludge zone as no plugging was observed in any of the tubes.

During treatment of the domestic wastewater the reactors were efficient enough to produce effluents with average total $BOD_5$ of 15 mg/L at HRTs ranging from 3-1 h. The average TSS in the effluent was 25 mg/L at 3 h and 2 h HRT. At 1 h HRT this was 60 mg/L. The permissible effluent $BOD_5$ and TSS for discharging the effluent into inland surface waters as specified by the Central Pollution Control Board (India) are 30 mg/L and 50 mg/L respectively. The observed efficiency in the reactors employed in the present investigation indicates the usefulness of this configuration for treatment of low strength wastewater. It is suggested that full-scale unit incorporating tube settler in the place of conventional GLSS may be tried for treatment of domestic wastewater.

# 1. INTRODUCTION

Thanks to the present day environmental consciousness that proper pollution control is a necessity, not an option. Even a stage has come when waste management systems efficient enough just to produce effluents that conform to standards stipulated by regulatory agencies are considered obsolete. This is reflected in the question that appeared as a news item: "Does anyone, including environmental experts and the plant authorities, ever question the permissible limits? Permissible for whom?" (Padte, 1995).

As waste management, in a way, is replacing the displaced resources, it is needless to say that biological methods are better suited for treatment of organic wastes. Considering the energy crisis that the present day world is facing, it is imperative to opt for treatment methods which are less energy intensive. This is especially so for developing countries. Anaerobic waste treatment methods, apart from the basic nature of low sludge production, enjoy a covetable energy budget. When aerobic methods demand a power requirement of 20-30 W, there is a useful energy production potential of 35 W in anaerobic process for an organic load of 1 kg COD/d (van Haandel and Lettinga, 1994).

Reactor configurations of yesteryears demanded high reactor volume to retain the slow growing methane formers as the Hydraulic Retention Time (HRT) and Biological Sludge Retention Time (BSRT) were same in these reactors. The various 'High rate' reactors developed during the

last two decades essentially aim at the retention of slow growing methane forming microorganisms independent of the HRT. The resulting higher biomass concentration, a better contact between the biomass and the waste organic matter and the higher waste stabilisation capacity per unit weight of the microbial film/granules that develop in the reactor have resulted in the improved loading capacity, process efficiency and stability in these reactors. The Upflow Anaerobic Sludge Blanket (UASB) reactors are the most popular among these "retained biomass" systems. Recent experience with full scale UASB reactors treating domestic wastewater demonstrates the usefulness of the process in municipal waste management in tropical regions (Gupta, 1992; Lettinga et al., 1993; van Haandel and Lettinga, 1994). However, this treatment system still suffers from certain limitations challenging its effectiveness especially for treatment of low-strength wastewaters.

Van Haandel and Lettinga (1994) reports that granulation has not been observed so far in any of the existing full-scale UASB reactors treating raw sewage. Achievement of loading rates conducive for granulation is restricted by the maximum superficial velocity that retain the flocculant seed sludge in the reactor. Though, experience from the field demonstrates that sludge granulation is not certainly a prerequisite for successful treatment of sewage in a UASB reactor, presence of granular sludge in the reactor can offer several benefits. The most important one is the improved specific waste stabilisation capacity of granular sludge due to the syntrophic association of different groups of the anaerobic microbial consortium.

Another limitation is that the low gas production rate during treatment of low-strength wastewater especially at low temperatures will impair sufficient bed expansion and mixing thereby affecting proper contact between the organic substrate and the microbes. Finally, the experience on full-scale UASB reactors indicates that a considerable fraction of the effluent COD is constituted by suspended organic matter. Thus, there is a need to modify the reactor configuration, with a special attention on the settler compartment so that the capturing of fine suspended organic matter can be maximised. The modified reactor should be capable of retaining the flocculant seed sludge in the reactor even at extremely low HRTs.

With this aim, a reactor configuration was conceived and designed by incorporating tube settlers in the settler zone replacing the conventional gas-liquid-solid separator of a UASB reactor. Two such bench-scale reactors with an empty bed volume of 9.16 litres each and settler compartment inclinations of $45^{o}$ and $60^{o}$ with the horizontal were employed for the studies on the following lines:

1. Development of granular sludge from flocculant type of seed sludge using sucrose based wastewater.
2. The performance evaluation of the reactors at different HRTs varying from 1-5 h while treating low-strength soluble and complex synthetic wastewaters.
3. Evaluation of the suitability of the reactor configuration to produce effluents conforming to standards while treating low-strength domestic wastewater.

4. Evaluation of the role of tube settlers on the performance of the reactors.

# 2. LITERATURE REVIEW

The "fire fairy of the marshes" very often appears in the myths and legends of many lands. The ancient civilisations of Egypt, China and Rome record the presence of a natural gas in the marshes and bogs which burns with a dancing pale-blue flame at dusk, giving rise to the tales of "Will-o'-the-wisp". The first person to attribute a scientific tint for the fairly-tale was van Helmont in 1630 by proving the burnability of "swamp-gas" (van den Berg, 1984). Later, Alexandre Volta in 1776 associated the formation of this 'combustible air' with decaying vegetation. This finding led to subsequent discoveries by Bechamp, Popoff, Tappeneiner, Hoppe Seyler, Söhngen and Omelianski which proved the microbial basis for the origin of methane gas (Sahm, 1984). Since then, the knowledge on microbiology and the biochemistry of anaerobic process developed slowly over a long period of time. As of now anaerobic treatment is considered to be a matured technology with a sound scientific and engineering foundation (Iza et al., 1991).

The biological conversion of waste into other products that are less polluting and offensive were already been employed since long for waste treatment: the septic tank, for example, was introduced by Mouras in 1860 (Van der Meer, 1979). However, an understanding that the water purification in such installation is effected by anaerobic microbial process, was shown by Groenewege in 1920. He was able to conceive even stages like liquefaction of the complex organic molecules to acids and alcohols and the subsequent conversion of these intermediary products to

methane. In addition, he speculated that the slow growth of the methane bacteria, observed in the laboratory, could cause the slow start-up of the septic tanks (Van der Meer, 1979).

Anaerobic waste stabilisation methods offer several advantages as compared to aerobic methods like low sludge production, higher loading potential since the process is not limited by the oxygen transfer rate at high oxygen utilisation rate and useful byproduct generation in the form of methane gas of high calorific value. Despite these advantages, the process could not gain user confidence till recently as anaerobic treatment units were infamous for their unreliability and high reactor volume requirement. The development of several novel reactor designs based on better comprehension of the microbiology and biochemistry of the process during the last two decades, has made the more customarily tagged advantages of the process a "reality"; this has brought the process back in reckoning as a viable and productive alternative in biological waste treatment of low, medium and high strength wastes.

## 2.1 Anaerobic Digestion Process Fundamentals

The anaerobic conversion of complex organic waste to $CH_4$ and $CO_2$ is a multistep process involving many interdependent, sequential and parallel reactions and require the participation of at least three different trophic groups of microorganisms which are linked by their individual substrate and product specificities. The pathways of anaerobic process are presented in Figure 2.1. Six distinct biochemical steps involved in the process according to Gujer and Zehnder (1983) are:

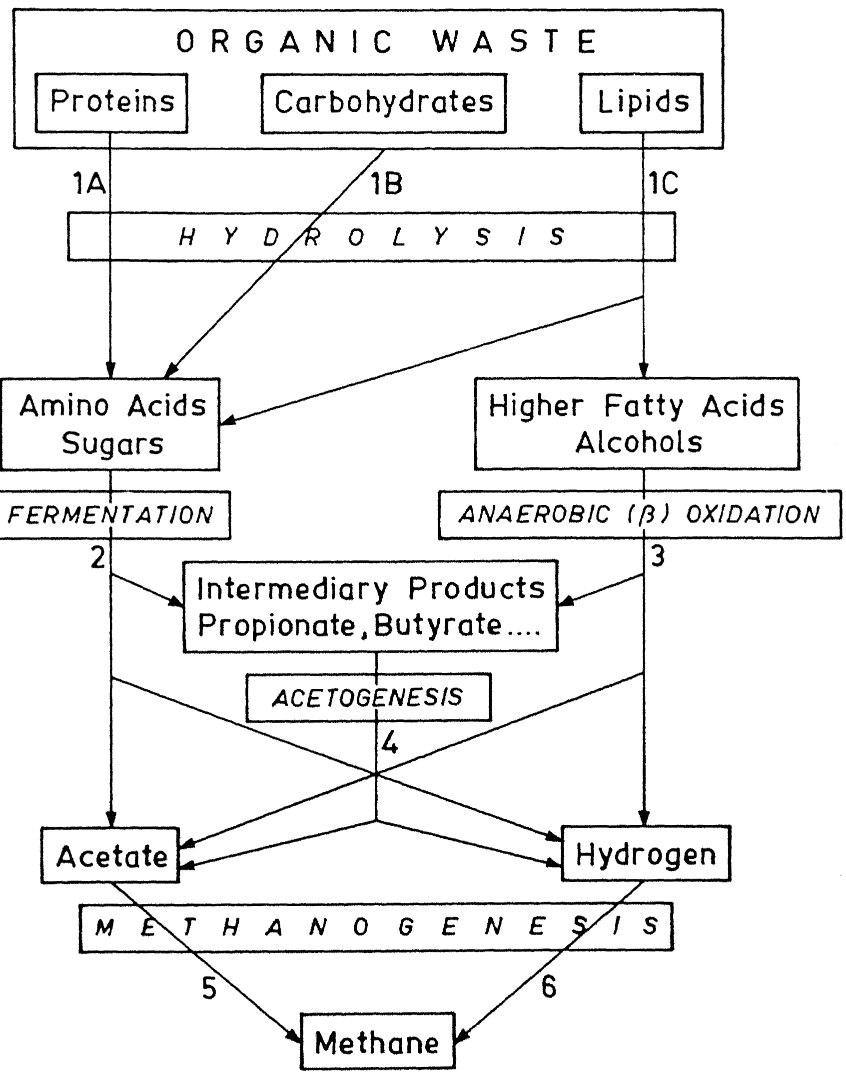



Fig. 2.1. Pathway of Anaerobic Biodegradation.
(Adapted from Kasper and Wuhrmann, 1978)

1. Hydrolysis of proteins, lipids and carbohydrates.
2. Fermentation of sugars and amino acids.
3. Anaerobic oxidation of long chain fatty acids and alcohols.
4. Anaerobic oxidation of intermediary metabolites.
5. Conversion of acetate to $CH_4$.
6. Conversion of $H_2$ to $CH_4$.

The first two processes are carried out by a group of microorganisms generally termed as acid formers. Anaerobic oxidation of long chain fatty acids and intermediary volatile fatty acids and alcohols to acetate are mediated by a second group collectively called as the Obligate $H_2$-Producing Acetogenic Bacteria (OHPA). Methanogenic bacteria synthesize methane from acetate as well as $CO_2$ and $H_2$. A coordinated participation of these three groups of bacteria is required for the complete stabilization of organic compounds.

**2.1.1 Hydrolysis of Particulate Organic Matter**

Organic polymeric materials must be rendered soluble (usually mono- or dimers) before they can enter the bacterial cell. This process of solubilisation is brought about by exocellular enzymes excreted by hydrolytic and acid forming bacteria. In terms of chemical composition, three groups of organics are considered as the major components of complex organics: carbohydrates, proteins and lipids.

Lignocellulosic materials (composed of cellulose, hemicellulose and lignin) are the most abundant natural organic compound. The hydrolysis products of cellulose are cellobiose (a dimer) and glucose whereas hemicellulose hydrolyses to pentoses, hexoses and uronic acids (Colberg,

1988). Lignin is highly recalcitrant and because of its association with cellulose, lignin decomposition is considered to be the rate-limiting step in the degradation of lignocellulosic materials.

Proteins are hydrolysed by exocellular enzymes, called proteases, into polypeptides and amino acids. According to Lackey and Hendrickson (1958), the general scheme of enzymatic protein breakdown proceeds through the following steps: protein ⟶ proteoses ⟶ peptones ⟶ peptides ⟶ amino acids. Peptones are the largest protein residues which can pass through a bacterial cell wall. Proteases are of two types: the extracellular, known as proteinases which attack the whole protein, and the intracellular, known as peptidases which cut amino acids off the end of proteins or polypeptides. Comparatively a few organisms are capable of excreting sizable quantity of proteinases into their environment and they require a readily utilisable nitrogen source for synthesis of the enzyme.

The degradation of lipids in anaerobic environment proceeds through the initial breakdown of fats by a group of esterases, called lipases, to their constituent long-chain fatty acids and glycerol moieties. Upon complete hydrolysis, phospholipids yield one equivalent of glycerol, one equivalent of phosphoric acid and two equivalents of fatty acids (Pavlostathis and Giraldo-Gomez, 1991).

The solubilisation of complex organics is a crucial step in the overall stabilisation of the organic matter as without hydrolysis organic carbon cannot enter a cell. Different types of polymers are degraded at different rates. Based on a comparative study of the

acid-phase anaerobic digestion of cellulose, starch and glucose, Noike et al. (1985) concluded that hydrolysis of cellulose was the rate limiting step. Kinetic studies conducted by Pavlostathis and Giraldo-Gomez (1991) on anaerobic digestion of waste activated sludge indicated that hydrolysis of particulate protein is the major rate-limiting step in the anaerobic digestion of biological sludge.

### 2.1.2 Fermentation of Sugars and Amino Acids

Fermentation can be described as a microbial process in which organic compounds serve both as electron donors and as electron accepters (Gujer and Zehnder, 1983). Sugars and amino acids formed during hydrolysis are taken up by the bacteria and fermented to intermediary metabolites like propionate, butyrate, lactate, ethanol, $H_2$, $CO_2$ and acetate. The hydrogen produced originates from dehydrogenation of pyruvate. According to Thauer et al. (1977) fermentation step is not inhibited by elevated partial pressure of $H_2$ (around 0.5 atm. $H_2$). However, the concentration of $H_2$ plays an important role in controlling the proportion of the various products formed by acidogenic bacteria. In mixed cultures where partial pressure of hydrogen is kept low by the action of $H_2$-utilising methanogenic bacteria, acetate, $H_2$ and $CO_2$ are the major end products of fermentation. When $H_2$ concentration increases because of some stress on the $H_2$-utilising methanogens, there is an increasing tendency for the formation of reduced products especially propionic, butyric and lactic acids as a means of disposing the "surplus hydrogen" generated.

Major bacterial genera taking part in this process have been identified as facultative anaerobes like *Streptococci* and *Enterobacteriaceae* as well as strict anaerobes like *Clostridia* and *Bifidobacteria*. The facultative anaerobes, apart from being associated with the production of intermediary metabolites also play an important role in the anaerobic microbial ecology. They utilize oxygen which may be initially present in the wastewater, thus leading to the reducing environment required for the fastidious methanogenic bacteria (Sahm, 1984).

The available data on growth kinetics for fermentation organics indicate that this reaction does not limit the performance of anaerobic digesters. This reaction is far from saturation and is not extremely pH dependent (Gujer and Zehnder, 1983).

### 2.1.3 Anaerobic Oxidation of Long- and Short-chain Fatty Acids

The degradation of long-chain fatty acids released from hydrolysis of fat has been reported to be through $\beta$-oxidation (Stronach, 1986). This is a cyclic process releasing one acetate unit per cycle and is repeated till the fatty acid is completely converted either to acetate, or propionate and acetate depending on whether the acid has even or odd number of carbons. The intermediary metabolites like butyrate, propionate, ethanol and lactate formed by acidogenic bacteria also have to be converted to acetate before methanogenesis. These conversions known as "anaerobic oxidation", as defined by Gujer and Zehnder (1983), is a microbial process in which molecular hydrogen is the main sink for electrons. Hydrogen is produced via the oxidation of

reduced NAD (P) and/or ferredoxin (a transfer of electrons to protons). The microorganisms mediating the oxidation of volatile acids to acetate is known as obligate hydrogen producing acetogenic bacteria (OHPA). A syntrophic (Syn-together, trophein-eat) relationship exists between this group and $H_2$ utilising methanogens. The synergistic association of the two species was reported by Bryant et al. (1967). Until then it was thought that the methanogenic bacteria use directly the products of acidogenic bacteria.

Reactions for acetate formation from the intermediary metabolites in the presence of OHPA are shown below (Harper and Pohland, 1986):

| Reaction | $\Delta G^{o}$/reaction (KJ) | |
|---|---|---|
| $CH_3CH_2COO^- + 3H_2O \longrightarrow CH_3COO^- + H^+ + HCO_3^- + 3H_2$ | +76.1 | [2.1] |
| $CH_3CH_2CH_2COO^- + 2H_2O \longrightarrow 2CH_3COO^- + H^+ + 2H_2$ | +48.1 | [2.2] |
| $CH_3CH_2OH + H_2O \longrightarrow CH_3COO^- + H^+ + 2H_2$ | +9.6 | [2.3] |
| $CH_3CHOHCOO^- + 2H_2O \longrightarrow CH_3COO^- + HCO_3^- + H^+ + 2H_2$ | -4.2 | [2.4] |

Proton is the sole terminal electron accepter and formation of molecular $H_2$ is the only way to regenerate the reduced coenzymes. Thermodynamically, all the reactions except 2.4 are endergonic. The equilibrium shift is towards the left and the organisms are unable to utilise organic substrates unless an extremely low partial pressure of $H_2$ is maintained in the environment. The partial pressure of $H_2$ cannot exceed about $10^{-4}$ atm. for energy to be available for propionate oxidising acetogens (Thauer et al., 1977). This low $H_2$ pressure can be maintained by an intensive cell contact between acetogenic and methanogenic bacteria (Thiele et al., 1988). Thus, a simultaneous

transfer of electrons from the acetogens to $H_2$ utilising methanogens is absolutely essential for growth of acetogens. This process termed as interspecies electron or $H_2$ transfer is a crucial step in anaerobic biotechnology. Some of the recent studies on the microstructure of anaerobic granules degrading soluble carbohydrate (McLeod et al., 1990; Fang et al., 1994) show for such symbiotic association of the two groups of microorganisms thus permitting reactions that yield energy for the growth of both species.

Formate had been identified as one of the intermediary metabolite formed during anaerobic fermentation (Harper and Suidan, 1991) and it was also known that many methanogens could utilize formate as carbon and energy source (Sahm, 1984). The mode of formate formation from pyruvate can be either in the presence of pyruvate-formate lyase or ferredoxin-$CO_2$ oxido reductase (Grobicki and Stuckey, 1989):

$$\text{Pyruvate} \xrightarrow[\text{formate-lyase}]{\text{Pyruvate}} \text{Acetate} + \text{Formate} \qquad [2.5]$$

$$\text{Pyruvate} + (\text{Ferredoxin})^{ox} \longrightarrow \text{Acetyl-CoA} + CO_2 + [\text{Ferredoxin}]^{red} \qquad [2.6]$$

$$2[\text{Ferredoxin}]^{red} + HCO_3^- + 2H^+ \xrightarrow[CO_2 \text{ oxidoreductase}]{\text{Ferredoxin}} \text{Formate} + H_2O + 2[\text{Ferredoxin}]^{ox} \qquad [2.7]$$

$$2[\text{Ferredoxin}]^{red} + 2H^+ \xrightarrow[\text{Hydrogenase}]{} H_2 + 2(\text{Ferredoxin})^{ox} \qquad [2.8]$$

The second pathway (reaction 2.6 and 2.7) is the major route for formate production. Hence formate is the electron sink product during

regeneration of reduced ferredoxin in bacteria which do not contain hydrogenase.

The first experimental evidence for the role of formate as an important extracellular intermediate in the conversion of ethanol to methane in anaerobic flocs was reported by Thiele and Zeikus (1988). They proposed a bicarbonate-formate electron shuttle mechanism to explain the electron flow during syntrophic ethanol conversion to methane by anaerobic flocs. In this model (Figure 2.2) $HCO_3^-$ is the substrate for syntrophic acetogens and formate is the electron carrier. Studies by Boone et al. (1989) based on the diffusion of $H_2$ and formate also showed that interspecies formate transfer may be the predominant mechanism of syntrophy. Grobicki and Stuckey (1989) reported the appearance of high levels of formate in anaerobic baffled reactors during shock loads, followed by a rapid reactor recovery indicating unusual stability. They propose that it may be desirable to encourage formate production through microbial selection, reactor design or environmental conditions. However, more work is needed to envisage the role of formate in anaerobic degradation of different wastewaters.

Syntrophic acetogens have not been physiologically well characterised. Among the two reported organisms of this group *Syntrophomonas wolfei* is a versatile anaerobic bacterium capable of converting octanoic, heptanoic, caproic, valeric and butyric acids to a mixture of acetic and propionic acid using $\beta$-oxidation with release of hydrogen or formate (McInerney et al. 1981; Boone et al., 1989). The generation time of this microorganism on butyrate is about three days.

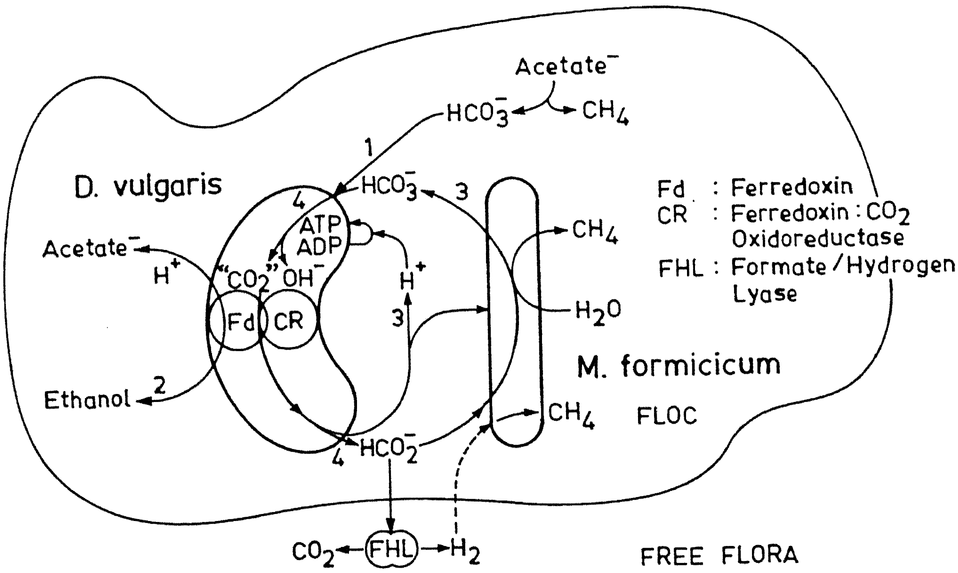



Fig. 2.2. Hypothetical Model Illustrating the Principles of the Bicarbonate-Formate Electron Shuttle Mechanism (Adapted from Thiele and Zeikus, 1988)

*Syntrophobacter wolinii*, a slow growing bacteria with a generation time of about seven days, can utilize only propionate and crotonate as its substrate (Boone and Bryant, 1980). Since crotonate is unlikely to occur in anaerobic digestor, propionate is the only available substrate for these organisms. Thus, population of *S. wolinii* in stable digesters may be limited. This can lead to the situation of "persistence of propionate" during the recovery period of such digestors after organic overloading. Thus accumulation of propionic acid can be considered as an indication of stress in anaerobic digestion system.

Reviewing kinetic studies using municipal sewage, reported in literature, Pavlostathis and Giraldo-Gomez (1991) conclude that as long as the digestor retention time is longer than the washout retention time for OHPA, lipid fermentation rates can be faster than the degradation rates of protein and carbohydrates.

### 2.1.4 Methenogenesis

Methane is formed through decarboxylation of acetate as well as by the reduction of $CO_2$ by $H_2$. The bacteria mediating these reactions are obligate anaerobes and require a redox potential of the order of -330 mV, which corresponds to one molecule of oxygen in about $10^{56}$ liters of water (Sahm, 1984). Although oxygen is the potent inhibitor of methanogenesis, methane-forming bacteria are not killed when exposed to high concentrations of oxygen. They survive and proliferate when environmental conditions permit them to do so.

Methanogens are the ultimate trophic group in the process of anaerobic digestion. Most of the species oxidize $H_2$ and reduce $CO_2$ to

form methane, as this reaction is thermodynamically the most favourable (Sahm, 1984). Many methanogenic bacteria are able to utilize formate as this reaction is also thermodynamically very favourable (Grobicki and Stuckey, 1989). Energy yielding reactions by methanogens are as follows (Daniels et al., 1984):

| Reaction | | | $\Delta G^{o}$/reaction (KJ) | |
|---|---|---|---|---|
| $4H_2 + CO_2$ | $\longrightarrow$ | $CH_4 + 2H_2O$ | - 138.8 | [2.9] |
| $4HCOOH$ | $\longrightarrow$ | $CH_4 + 3CO_2 + 2H_2O$ | - 119.5 | [2.10] |
| $CH_3COOH$ | $\longrightarrow$ | $CH_4 + CO_2$ | - 27.6 | [2.11] |
| $4CH_3OH$ | $\longrightarrow$ | $3CH_4 + CO_2 + 2H_2O$ | - 310.5 | [2.12] |
| $4CH_3NH_3^+ + 2H_2O$ | $\longrightarrow$ | $3CH_4 + CO_2 + 4NH_4^+$ | - 225.7 | [2.13] |

Although very few species degrade acetate to $CH_4$ and $CO_2$, 70% methane generated in anaerobic digesters originate via the methyl group of acetate. *Methanosarcina barkeri*, *Methanosarcina vacuolata*, *Methanococcus mazei* and *Methanothrix soehngenii* are well-known acetate-degrading methanogens (Sahm, 1984; Pohland, 1992). As the energy available from acetate is very low, these organisms grow very slowly. *M. mazei* and *M. barkeri* are among the most versatile methanogenic bacteria as they can utilize methanol and methylamines in addition to acetate and $H_2$.

Among the well studied species converting acetate to methane, *M. Barkeri* strains grow much faster on acetate with a generation time of 2-3 days and have a saturation substrate concentration ($K_s$) value of 5 mM $L^{-1}$ for acetate. On the other hand, *M. soehngenii* has a generation

time of 10 days or more, but has a relatively low $K_s$ value of 0.7 mM $L^{-1}$. Thus these can out-compete other strains at longer detention times which are generally achieved in high rate anaerobic reactors (Sahm, 1984).

The formation of methane, the most reduced form of carbon from $CO_2$ probably requires four reduction steps involving the oxidation levels of formate, formaldehyde, methanol and methane. These intermediates have not been isolated in free form and thus appear to be bound to different carriers during the reduction process. Energy released during these reactions, is utilized for their growth. No respiratory electron chain has been identified in any methane bacteria till now.

All methanogens examined to date (with the exception of *Methanothrix species*) are capable of $CH_4$ formation by oxidation of $H_2$ (Sahm, 1984; Harper and Pohland, 1986; Pohland, 1992). The efficient removal of $H_2$ produced during the fermentation of carbohydrates and proteins and the anaerobic oxidation of fatty acids by methanogens allow the aforementioned reactions to proceed under natural and physiological conditions. Although only about one third of the methane produced in anaerobic digestors come from the reduction of $CO_2$ by $H_2$, this step involving the interspecies $H_2$ transfer and utilisation is far more important since it regulates the rate of $H_2$-acetate producing reactions.

Methanogens differ from other bacteria in several biochemical characteristics (Sahm, 1984; Zehnder, 1978). Muramic acid, a characteristic cell wall component of bacteria, is absent in methane bacteria. Methanogens except *Methanobacteriaceae* and

*Methanobrevibacteriaceae* possess envelops composed of glycoprotein or protein subunits. Therefore these bacteria are resistant to the cell wall active antibiotics such as penicilline, D-cycloserine, Vancomycin and Cephalosporin. Composition of lipids differ markedly from that of other typical bacteria. Coenzyme M, coenzyme $F_{420}$ and coenzyme $F_{430}$ are found only in methanogens. These characteristics have led to the classification of methanogens as members of *Archaebacteria*, which is an ancient group and phylogenetically distinct from typical procaryotes. Other members of this group, in addition to methanogens, are extreme halophiles and thermo-acidophiles (Sahm, 1984).

### 2.1.5 Role of Sulfate Reducing Bacteria (SRB) in Methanogenesis

Extensive work has been done on the effect of sulfate on methanogenesis. SRB play a dominant role in the overall stability of anaerobic digesters as they can use the same energy source as methanogens, namely acetic acid and $H_2$ (Isa et al., 1986; Karhadkar et al., 1987; McCartney and Oleszkiewicz, 1991; Harada et al., 1994). They can be considered as the most effective $H_2$ scavengers . Further SRB can convert propionate to acetate through an energetically favourable reaction. In all these reactions, given below, sulfate acts as electron accepter (Harper and Pohland, 1986; Moletta et al., 1986):

| Reaction | | $\Delta G^o$/reaction (KJ) | |
|---|---|---|---|
| $4H_2 + SO_4^{=} + H^+$ | $\longrightarrow HS^- + 4H_2O$ | -151.9 | [2.14] |

$$CH_3COO^- + SO_4^{=} + H^+ \longrightarrow 2HCO_3^- + H_2S \qquad -59.9 \qquad [2.15]$$

$$4CH_3CH_2COO^- + 3SO_4^{=} \longrightarrow 4CH_3COO^- + 4HCO_3^- + 3HS^- + H^+ \qquad -151.0 \qquad [2.16]$$

The reduction of sulfate to sulfide yields more energy than methanogenesis making the latter noncompetitive. However, methanogenesis and sulfate reduction are not mutually exclusive and in presence of excess $H_2$ have no effect on each other (Stronach et al., 1986). However, the product formed by SRB, i.e. $H_2S$ is toxic to methanogens at higher concentration. The role of SRB in anaerobic methanogenesis has been summarized as follows (McCartney et al., 1990): (a) generate sulfide that may result in product inhibition or toxicity to methanogens, (b) increases the reactor pH, which is more favourable for methanogens, (c) competes with methane producers for acetate and $H_2$ and (d) accelerates the degradation of organics like propionate.

## 2.2 Thermodynamic and Kinetic Interactions

From the above brief discussion on microbiology and biochemistry of anaerobic methanogenesis, it can be seen that the stabilisation of complex organics is a multistep process and the microbial consortium mediating the process effects many interdependent, consecutive and parallel reactions. Most of the attempts to kinetically describe this complex process relies upon the so-called rate-limiting step approach (Pavlostathis and Giraldo-Gomes, 1991). In this approach the kinetics of the slowest step is considered to govern the overall waste stabilisation rate.

It may be more appropriate to state that in an anaerobic process the rate-limiting step is dependent on the nature of substrate, process configuration and loading rate. The hydrolysis of insoluble biopolymers has been identified as the overall rate limiting step in the anaerobic digestion of sludge and particulate organics. Rates of monomer formation from such polymers depend upon their structure, substituent groups and the types of organisms present in the digester (Speece, 1985). The complex structure of natural cellulose and the lignin sheath encapsulating it make the solubilisation of this substrate among carbohydrates, the rate-limiting step. However, the rate of hydrolysis of protein under anaerobic condition is found to be slower than that of carbohydrates (Pavlostathis and Giraldo-Gomes, 1991). With simple substrates, conversion of acetate to methane is identified as the rate limiting step (Speece, 1985; Henze and Harremoes, 1985).

Experimental evidence indicates that the rate-limiting step is dependent on the loading rate also. The hydrolysis is found to be the rate-limiting step at conventional loading rates during stabilisation of municipal sludges. The rate of hydrolysis determines, for a given retention time, the potential maximum substrate concentration possible for methanogens, which in turn determines their maximum possible specific growth rate. Thus hydrolysis affects the overall process kinetics. However, as the loading rate increases or sludge retention time reduces, the methanogenesis from acetate becomes the rate-limiting step (Pavlostathis and Giraldo-Gomes, 1991).

The harmony among propionate oxidation, acetate decarboxylation and $H_2$ oxidation is crucial for anaerobic stabilisation of organic compounds. Existence of such a harmony in anaerobic granules, a specialty of certain reactor configurations, by the juxtapositioning of the OHPA and hydrogen consuming methanogens which enhances interspecies electron transfer efficiency was suggested by Thiele et al. (1988). Some of the recent studies on the microstructure of anaerobic granules stabilising complex wastewater evidences the development of syntrophic microcolonies of these two groups of organism around the acetoclastic *Methanothrix* central core (MacLeod et al., 1990; Fang et al., 1994). Reactor configurations capable of developing such microbial flocs/granules exhibit high rates of acetogenesis and methanogenesis leading to high specific waste stabilisation capacity.

## 2.3 Kinetic Models

Anaerobic treatment, from the kinetics viewpoint, may be described as a three step process involving (a) hydrolysis of complex material, (b) acid production and (c) methane production.

The most commonly applied model for the description of the hydrolysis rate in anaerobic system is first-order with respect to the concentration of degradable particulate organic matter (Pavlostathis and Giraldo-Gomes, 1991):

$$\frac{dF}{dt} = - k_h F \qquad [2.17]$$

where $k_h$ = hydrolysis rate constant ($T^{-1}$)

$F$ = degradable particulate organic matter ($ML^{-3}$).

For a batch reactor, integration of the above equation leads to:

$$F = F_o e^{-k_h t} \qquad [2.18]$$

where $F_o$ = concentration of degradable particulate organic matter at time, t = 0 ($ML^{-3}$).

For a CSTR at steady-state, the following equation is obtained

$$F = \frac{F_o}{1 + k_h \theta_h} \qquad [2.19]$$

where $F_o$ and F = influent and effluent concentration of degradable, particulate organic matters, respectively ($ML^{-3}$).

$\theta_h$ = hydraulic retention time (T).

Acetogenesis and methanogenesis can be successfully modeled using Monod kinetics. Monod (1949) has described the effect of growth-limiting substrate concentration on the rate of microbial growth by the equation

$$\mu = \mu_m \frac{S}{K_s + S} \qquad [2.20]$$

where,

$\mu$ = specific growth rate of biomass ($T^{-1}$) which can be defined as $(\frac{dx/dt}{x})$ where x is the concentration of biomass present ($ML^{-3}$)

$\mu_m$ = maximum specific growth rate ($T^{-1}$)

S = residual growth-limiting substrate concentration ($ML^{-3}$)

$K_s$ = half-saturation constant numerically equal to the substrate concentration at which $\mu = \mu_m/2$ ($ML^{-3}$).

Lawrence and McCarty (1969) relate the rate of substrate utilisation to the concentration of microorganism in the reactor and to the concentration of substrate surrounding the organism by the equation

$$\left(\frac{ds}{dt}\right) = \frac{kxS}{K_s + S} \qquad [2.21]$$

where,

$\left(\frac{ds}{dt}\right)$ = overall substrate utilisation rate ($ML^{-3}\ T^{-3}$)

k = maximum specific substrate utilisation rate ($T^{-1}$)

S = substrate concentration surrounding the biomass ($ML^{-3}$)

$K_s$ = half-saturation constant, which has a value equal to the substrate concentration when $\left(\frac{ds/dt}{x}\right) = k/2$ ($ML^{-3}$)

x = active biomass concentration ($ML^{-3}$).

Equation [2.21] can be written as

$$q = \frac{ks}{K_s + S} \qquad [2.22]$$

where,

q = specific substrate utilisation rate ($T^{-1}$) which can be defined as $\left(\frac{ds/dt}{x}\right)$.

Again, an expression which can be used to describe the net growth rate of microorganism in a completely mixed continuous anaerobic treatment system can be written as follows (Lawrence and McCarty, 1969)

$$\mu = Y q - K_d \qquad [2.23]$$

where,

Y = growth yield which is mathematically defined as $\frac{dx}{ds}$

$K_d$ = microorganism decay coefficient ($T^{-1}$).

Numerous investigators have studied the kinetics of anaerobic degradation of complex biopolymers, soluble carbohydrates, fatty acids and amino acids under a variety of operational conditions. These studies have included fundamentals of the biochemistry of methanogenesis as well as the substrate conversion rates associated with various process configurations. Pavlostathis and Giraldo-Gomes (1991) have reviewed some of these studies and have indicated that variances in kinetic parameters for both enriched and mixed culture were largely accountable to the method of measurement, process configuration and operation employed. However, the ranges of derived kinetic constants are informative and can provide a basis for process selection, design and control.

Gujer and Zehnder (1983) have calculated the apparent first order rate constant from literature data for the hydrolysis of biopolymers. These values for various biopolymers are listed in Table 2.1. These hydrolysis rate constants also include the cummulative effect of all the microscopic processes like particle deposition, entrapment and sorption that effectively stunt substrate utilisation (Pohland, 1992). All particulate materials are not degraded at equal rates. Factors like surface area-to-volume ratio and the constituent polymer of the particle affect the rate of hydrolysis. The flow dynamics resulting from a particular operational configuration is another factor which greatly influences the substrate solubilisation.

Table 2.1: Hydrolysis of biopolymers under anaerobic condition (adapted from Gujer and Zehnder, 1983)

| Biopolymer | First order hydrolysis rate constant, $K_h$(1/d) | Temperature, ($^oC$) |
|---|---|---|
| Lipids | 0.08 - 1.7 | 34 - 40 |
| Proteins | 0.02 - 0.03 | 34 - 35 |
| Cellulose | 0.04 - 0.13 | 34 - 35 |
| Hemicellulose | 0.54 | 35 |

Kinetic data based on Monod kinetics for anaerobic fermentation of various substrates was compiled by Pavlostathis and Giraldo-Gomes (1991). The kinetic constants pertaining to each sub process are given in Table 2.2.

**2.4 Effect of Mass Transfer on Kinetics**

The success or failure of anaerobic waste treatment process is largely dependent on the transport of sustaining substrate to the microorganisms and its availability to provide energy for growth and metabolism. First, the substrate must be transported from the bulk liquid across a stagnant liquid layer in the proximity of the biofilm and then further transported to the surface of the aggregate. This step is commonly termed "external mass transport". Then the substrate must diffuse throughout the aggregate matrix where it will be catabolized. Intermediate products diffuse and react inside the aggregate, while final products must diffuse out to the aggregate surface and finally to the bulk liquid (Pavlostathis and Giraldo-Gomes, 1991). The efficiency

Table 2.2: Summary of values of kinetic constants for various substrates utilized in mesophilic anaerobic treatment processes (adapted from Pavlostathis and Giraldo-Gomes, 1991)

| Substrate | Process | k (g COD/ g VSS-d) | $K_s$ (mg COD/L) | $\mu_m$ (1/d) | Y (g VSS/ g COD) | $K_d$ (1/d) |
|---|---|---|---|---|---|---|
| Carbohydrates | Acidogenesis | 1.33-70.6 | 22.5-630 | 7.2-30 | 0.14-0.17 | 6.1 |
| Long-chain fatty acids | Anaerobic oxidation | 0.77-6.67 | 105-3180 | 0.085-0.55 | 0.04-0.11 | 0.01-0.015 |
| Short-chain fatty acids (except acetate) | Anaerobic oxidation | 6.2-17.1 | 12-500 | 0.13-1.20 | 0.025-0.047 | 0.01-0.027 |
| Acetate | Acetoclastic methanogenesis | 2.6-11.6 | 11-421 | 0.08-0.7 | 0.01-0.054 | 0.004-0.037 |
| Hydrogen/carbon dioxide | Methanogenesis | 1.92-90 | $4.8 \times 10^{-5}$-0.60 | 0.05-4.07 | 0.017-0.045 | 0.088 |

of the external mass transfer largely depends on the contact opportunity provided at the microb-substrate interface. For soluble substrate, convection and diffusion are the main mechanisms of transport whereas for particulate substrate, two additional mechanisms, namely gravity sedimentation and interception come into play. These factors are influenced by the flow dynamics resulting from a particular operational configuration.

The internal mass transport of solute within a microbial matrix is normally approached and modeled using Fick's law (Pavlostathis and Giraldo-Gomes, 1991). The diffusion coefficient of the substrate is not an easily measurable parameter. However, the available information suggests that they are between 10% and 30% of those observed in clear water. The generation and temporal and spatial residence of gas bubbles within and on the surface of the microbial aggregates are suspected to dramatically disturb and alter substrate diffusion patterns. It is also expected to influence the configuration of the microbial conglomerate with respect to its structural fabric and relative porosity. Continuous evolution of gas through the gas channels may act as significant barrier to solute transport within the aggregate. However, these gas channels can act as an efficient drainage structure for gaseous products from the deep part of aggregate to the surface.

The mass transfer limitations are reported to influence the process kinetics and substrate utilisation rate significantly. Pavlostathis and Giraldo-Gomes (1991) report that there exists an apparent half-saturation constant which is greater than the intrinsic one and it

would increase as the mass transfer limitation becomes more severe. Such a finding would bring into question the absolute validity of recorded half-saturation concentration (Table 2.2) for exogenously supplied hydrogen, since they would likely be overestimated compared to substrate- (propionate) generated hydrogen. Now, one could suspect the existence of mass transfer limitations during the experiments of Contois (1959) in which half-saturation constant was found to be proportional to the influent substrate concentration.

Because of the complex energetic relationship involved in the anaerobic process, the concentration gradient of the intermediary metabolites between the bacterial population may have important effects on the behaviour of the microbial ecosystem. The close association of the microbial group favour interspecies metabolite transfer because of the minimisation of interspecies diffusional gradient for intermediate metabolites. In the case of $H_2$ formed by the Obligate Hydrogen Producing Acetogens (OHPA), such a close association avoids the gas-to-liquid mass transfer limitation (MacLeod et al., 1990; Fang et al., 1994). Thus microbial aggregation is beneficial for acetate production from higher acids as well as acetate removal by methanogens thus enhancing the specific waste stabilisation rate.

Recently there have been several reports on the existence of threshold for substrate uptake by methanogens. A threshold is considered to be the concentration of substrate below which the substrate consumption stops. This phenomenon has an important implication for the minimum COD that can be obtained by anaerobic

digestion (Pavlostathis and Giraldo-Gomes, 1991). This is especially true for acetate. For *Methanosarcina* and *Methanothrix* species, threshold values between 15-130 mg/L and 0.4-4mg/L respectively are reported in the literature. It is likely that the mass transfer limitations can result in a bulk liquid substrate concentration higher than the intrinsic threshold value in the case of externally supplied substrate. However, the threshold acetate concentration reported for granular sludge is 0.25 mg/L (Pavlostathis and Giraldo-Gomes (1991). It may be argued that acetate is internally produced and so the mass transfer limitations are minimised which will result in lower bulk liquid acetate concentration.

## 2.5 Factors Affecting Anaerobic Organic Stabilisation

Anaerobic digestion is affected by many factors among which, temperature, pH, nutrients and presence of toxicants/inhibitors in the wastewater to be treated, are the most important. They profoundly affect the microbial growth rate thereby affecting substrate utilisation and overall efficiency of treatment.

### 2.5.1 Temperature

Effect of temperature on the rate of anaerobic digestion dictates that this should be considered as the principal design parameter. Methanogenesis rate with suspended microbial system gradually increases from $25^{o}C$ with an optimum between $35-40^{o}C$ beyond which a decrease is observed as methane formers grow best in mesophilic temperature range (Zehnder, 1978). With retained biomass reactors, start-up will be prolonged at lower temperatures ($<25^{o}C$). However, once

developed, they may be operated at low temperatures (Stronach et al., 1986). Rapid alterations in temperature, even by few degrees, can result in a marked effect on microbial metabolism and may require several days for recovery. Inefficiencies of climatically heated small anaerobic digesters can be ascribed to diurnal temperature fluctuations. The change in operating temperature especially towards lower side, if it is required, should be effected gradually. This permits the microbial adaptation to the new temperature. It is observed that slow growing methanogenic bacteria adapt slowly to changed temperature conditions as compared to other groups present in anaerobic digesters. This results in rapid accumulation of volatile acid intermediates, which are inhibitory to methanogens leading to process failure.

### 2.5.2 pH and Alkalinity

Optimum pH range for methanogenesis has been reported to be between 6.8 and 7.4 (Zehnder, 1978). Low pH are particularly detrimental to methanogens than fermentative bacteria. Important buffering system present in anaerobic reactors is bicarbonate although ammonia may be predominant in the digestion of sludge and treatment of protein rich wastewaters. Under stable operating conditions, $HCO_3^-$ levels are reduced in the acidogenic phase and are released during methanogenesis. However during overloading, formation of high concentrations of volatile acids as well as subsequent inhibition of methanogenesis cause the decrease in pH. As the fermenting bacteria can continue to produce fatty acids despite pH depression, the environmental conditions further deteriorates and thus leading to the situation of

'Stuck digesters'. For the optimum performance of anaerobic digesters, ratio of volatile fatty acids to total alkalinity should be maintained around 0.1 (Sahm, 1984). Addition of alkalinity in the form of lime or $NaHCO_3$ may be required during the treatment of some industrial wastewaters. It is to be mentioned that pH adjustment alone cannot revive the performance of stuck digesters. A fresh active biomass addition may also be required.

### 2.5.3 Nutrients

Apart from organic carbon source, anaerobic microorganisms require nitrogen, phosphorus and other trace elements for cell synthesis. While domestic wastewater may contain all these components in required quantities, many industrial wastewaters lack some of them and thus have to be supplemented. The theoretical minimum COD/N/P ratio required for high loaded anaerobic process (0.8-1.2 g COD/g VSS.d) is around 400/7/1. For low loaded digesters, COD/N ratio increases dramatically to 1000/7 (Henze and Harremoes, 1985). Four elements, iron, cobalt, nickel and sulphur have been shown to be obligatory nutrients for methanogens converting acetate to methane (Speece et al., 1983; Takashima and Speece, 1990). Addition of iron and cobalt salts enhanced methane production even from domestic wastewater sludges and cattle manure which are generally assumed to be nutritionally adequate. Ni is a component of coenzyme $F_{430}$ present specifically in methanogens. Although high levels of sulphide adversely affect methane production, it is an essential nutrient for methanogens (Speece, 1983). Unionised $H_2S$ sulfide concentration required for optimal growth of methanogens is 13

mg/L and this corresponds to approximately 0.5% $H_2S$ in the head gas at equilibrium. It has been reported that sulphur content of methanogens is of the same magnitude as that of phosphorus and is in the range of 5-12 mg/g dry weight (Takashima and Speece, 1990).

**2.5.4 Inhibition and Toxicity**

It is generally observed that acetogens and methanogens are more sensitive to the presence of inhibitors as compared to fermentative bacteria. Inhibitors can be broadly categorised into three groups: (a) toxic compounds present in wastewater. They include heavy metal ions, pharmaceuticals, insecticides, sulphates and organic solvents. (b) components, which are essential nutrients in trace amounts, but are inhibitory at higher concentrations, e.g. sulphide. (c) obligate metabolic intermediates of the process acting as inhibitors at high concentrations. Propionate and butyrate are intermediary metabolites in the anaerobic degradation of organics, which are inhibitory at high concentrations. In an efficiently operating anaerobic system, where partial pressure of $H_2$ is maintained at very low level, formation and degradation of these volatile acids are well balanced. Overloading, decrease in hydraulic detention time or abrupt changes in temperature results in a stress on slow growing methane formers and leads to a rapid accumulation of volatile fatty acids. They are potential inhibitors of methane formers. Although the mechanism of inhibition is still obscure, undissociated volatile fatty acids seem to play an important role and thus the extent of inhibition is pH dependent. Propionate has been reported to be the most toxic among three volatile fatty acids to $H_2$

utilizing methanogens (Stronach et al., 1986). This inhibition, in turn, results in the increase in $H_2$ partial pressure of the system leading to the inhibition of acetogenic bacteria. Thus the accumulation of volatile fatty acids ultimately results in 'Stuck digesters'.

## 2.6 High Rate Anaerobic Reactors

Despite the well recognised advantages of anaerobic biological process, its application was restricted to the treatment of concentrated wastes such as animal refuse and the sludge derived from aerobic treatment processes. This was mainly due to the reactor design which employed suspended microbial system, where microorganisms were removed from the reactor along with the treated effluent. This necessitated a long residence time of even upto 20-30 days so as to keep high concentrations of the slow growing acetogenic and methane bacteria in the reactor, thus demanding large volume reactors. This restricted the process application to industrial and domestic wastewater treatment.

During last 2-3 decades, high rate reactor types which separate hydraulic retention time (HRT) from biological sludge retention time (BSRT) and allow the slow growing anaerobic bacteria to be retained within the reactor independent of wastewater flow, have been developed (Iza et al., 1991). Most of these high rate systems are essentially 'retained biomass systems', where microorganisms are present in the reactor as an attached biofilm grown on a support medium or as flocs/granules which are easily separable from aqueous phase. Using these reactor designs, it has been possible to operate the reactors at higher organic loading rates and at low HRTs. Increased efficiency of

these reactor configurations can be attributed to higher biomass concentration as well as juxtapositioning of different trophic microbial groups in a biofilm or granule resulting in better specific waste stabilisation capacity (Thiele et al., 1988). The introduction of these reactor designs has now made the application of anaerobic biotechnology to dilute and high strength organic wastewaters possible.

Figure 2.3 shows different configurations of high rate anaerobic wastewater treatment systems (van Haandel and Lettinga, 1994). Some of their salient features are briefly discussed here.

### 2.6.1 Anaerobic Contact Process

This was one of the first retained biomass reactor configuration to be introduced (Schroepfer et al., 1955; Coulter et al., 1957). In this system, microbial flocs are separated from treated effluent in an external gravity or centrifugal separating device and returned to the reactor. This is sometimes referred to as anaerobic activated sludge. Settleability of the microbial flocs depend upon waste characteristics and the loading rate. Higher loading rate may cause the buoyancy in the floc leading to poor settling. Degassification may become necessary in some instances (Sahm, 1984). Thus the process has a built-in maximum loading rate. The reactor design is especially suited for wastes having a certain amount of hard-to-digest solids that settle readily or attach themselves to settleable solids. These solids will then have a detention times well in excess of HRT and thus have a chance to be degraded (van den Berg, 1984). The process will not be affected by substrate diffusion

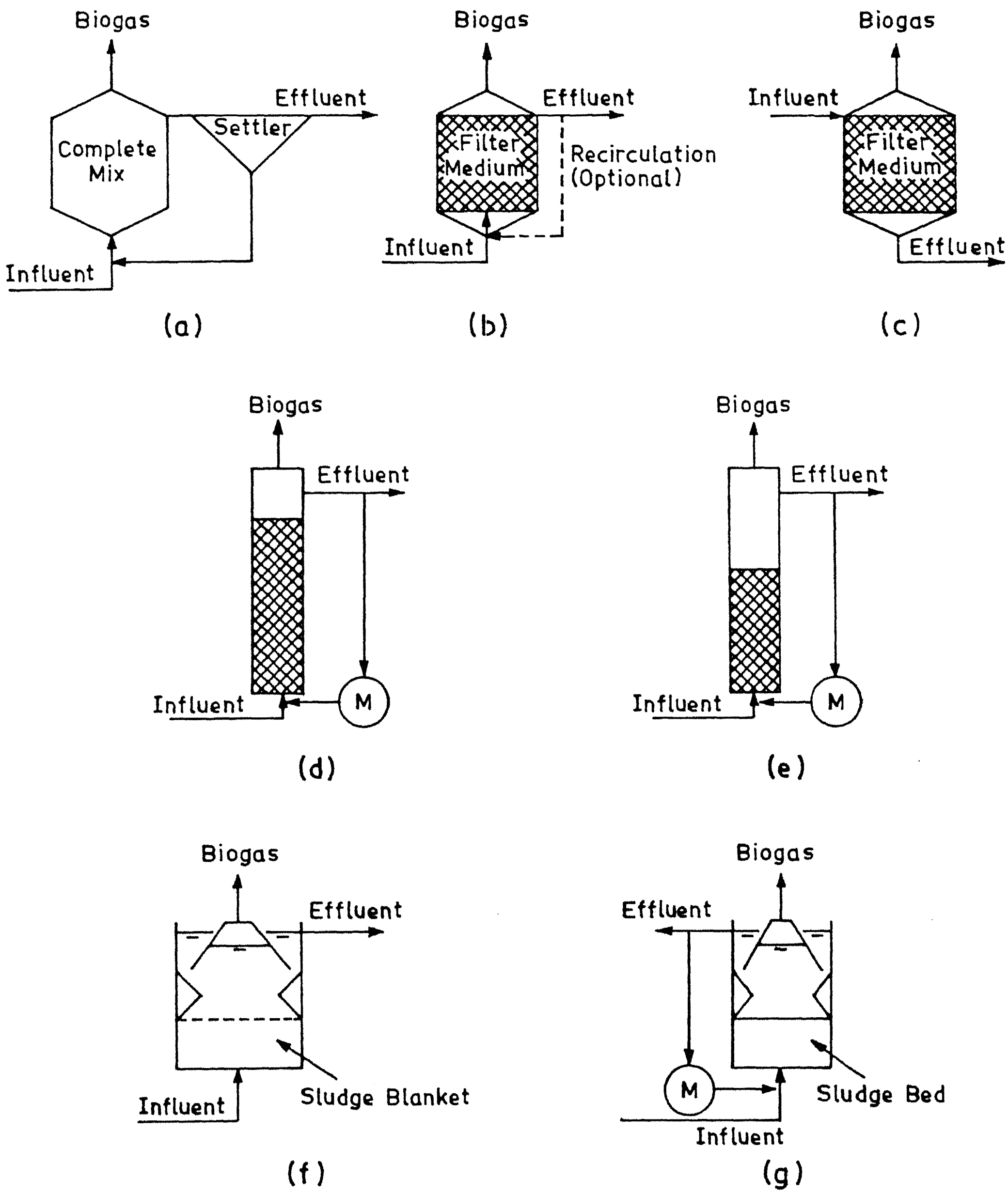



Fig. 2.3. Different Configurations of High Rate Anaerobic Waste-Water Treatment Systems. (Adapted from Lettinga and Van Haandel, 1994)
(a) Contact Process (b) Upflow Anaerobic Filter (c) Downflow Anaerobic Filter (d) Fluidised Bed (e) Expanded Bed (f) Upflow Anaerobic Sludge Blanket (UASB) Reactor (g) Expanded Granular Sludge Blanket (EGSB) Reactor.

limitation as mixing is provided in the reactor. However, the retained biomass concentration is comparatively less, thus limiting the loading rate.

### 2.6.2 Anaerobic Filter (AF)

The anaerobic filter was introduced by Young and McCarty in 1969 on the basis of earlier work by Coulter et al. (1957). Inert support materials in the form of sheets, rings and sphere are provided in the reactor either in random or ordered configuration for microbial attachment and biofilm development. The reactor may be operated in the upflow or downflow mode. Suspended microorganisms tend to collect at the bottom of the reactor. The process is suitable for dilute as well as high strength waste with easily degradable suspended material (van den Berg, 1984). It has been shown that organic loads upto 10-20 kg $COD/m^3.d$ can be applied when the concentration and nature of organic matter are favourable (van Haandel and Lettinga, 1994). Accumulation of suspended biomass and waste suspended solids along with the inorganic material precipitated from the waste ($CaCO_3$, metal sulphide etc.) cause plugging which leads to short circuiting and dead zone formation. This limits the specific microbial loading capacity (Speece, 1985). AF is capable of handling hydraulic and organic shock loads due to less possibility of microbial washout. The system has the added advantage that it can be satisfactorily operated without skilled supervision. Full-scale AF systems have been reported for treating various types of industrial wastewaters (van den Berg, 1984), but for sewage treatment

the system is rarely used at large scale (van Haandel and Lettinga, 1994).

### 2.6.3 Anaerobic Fluidised and Expanded Bed Reactors (FB/EB)

In this reactor type, fine carrier particles are used for the microbial film development. These particles with their attached biofilm are fluidised by high upflow liquid velocities derived by a combination of the influent and recirculated effluents. When the resulting bed expansion is upto 30%, the reactor is named as EB and if it is more than 30% it is named as FB reactor (Kosaric and Blaszczyk, 1990). Reactor performance depends on the evenness of the upflow of the liquid and hence the liquid distribution is very critical (Jewell, 1982). Advantages of FB reactor configuration are large surface area of the particles for biomass attachment, high concentrations of biomass attached to dense particles which cannot be easily washed out, the initial dilution of the influent with treated effluent minimising the organic shock loads and no plugging, channeling or gas hold up (Iza, 1991). However, the initial dilution of the influent with the recirculated effluent will be effected as a limitation to the reactor configuration for its applicability for treatment of low-strength waste like domestic sewage as it reduces substrate concentration gradient. So far, there are no full-scale installations for sewage treatment using FB/EB system, but a few pilot and bench-scale studies are reported (van Haandel and Lettinga, 1994).

### 2.6.4 Upflow Anaerobic Sludge Blanket (UASB) Reactor

The UASB reactor was developed in the 1970s by Lettinga and

his group at the University of Wageningen in the Netherlands (van Haandel and Lettinga, 1994). Figure 2.4 is a schematic representation of the UASB reactor with its characteristic devices. The process relies on the tendency of anaerobic bacteria to form flocs or granules that are retained within the reactor by an efficient gas liquid solid separation (GLSS) device located at the top of the reactor (Lettinga et al., 1980). This device divides the reactor into a lower part, the digestion zone, and an upper part, the settling zone. The wastewater is introduced as uniformly as possible over the reactor bottom. It then flows upwards through a blanket of anaerobic well settling sludge in the digestion zone. The organic matter present in wastewater is degraded by microbes present in this zone with the production of biogas. The treated liquid, biomass and the biogas rises up. Some of the sludge flocs that rise up with adhering gas bubbles will settle as the gas bubbles are released to the gas phase at the liquid-gas interphase under the GLSS. The deflector placed beneath the aperture of the gas collector units prevents the biogas bubbles from entering the settling zone. The space between two GLSS surface acts as the settling zone. The treated liquid enters into the settling zone via the aperture between the GLSS and deflector. The inclined walls of GLSS provide increased area for liquid flow so that settling velocity decreases as it approaches the water surfaces. The biomass escaping with the effluent settles in the settling zone and slides down the slope of the GLSS back into the digestion zone to become, once again, part of the sludge mass that digest the influent organic matter. Effluent from the settling zone is

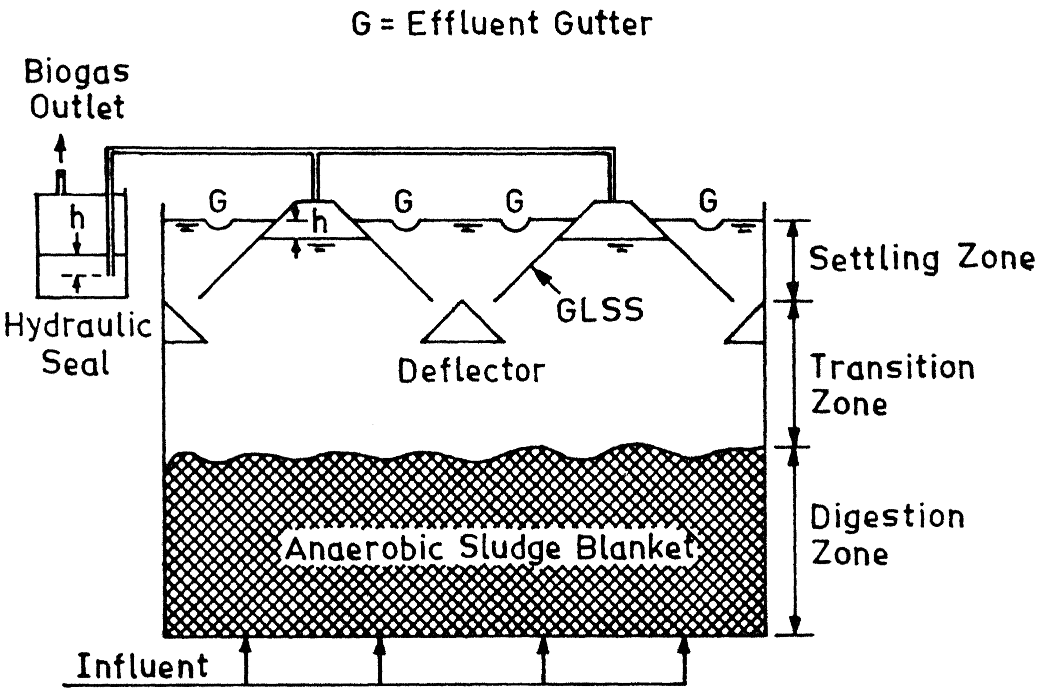



Fig. 2.4. Schematic Representation of an Upflow Anaerobic Sludge Blanket (UASB) Reactor.
(Adapted from Van Haandel and Lettinga, 1994)

collected initially in gutters provided on both sides of the settling zones. These gutters empty the effluent into the main outlet channel. The presence of a GLSS on top of the digestion zone enables the system to maintain a large sludge mass in the UASB reactor while an effluent free of suspended solids is discharged.

The UASB reactor is the most widely used high rate anaerobic system for anaerobic sewage treatment. Several full-scale plants have been put into operation in tropical and subtropical conditions and many more are presently under construction (van Haandel and Lettinga, 1994). Reactors operating at 4-6 h HRTs on domestic wastewaters have exhibited COD and BOD (total/total) removal efficiencies in the range of 50-70% and 70-90% respectively (Lettinga, 1992).

### 2.6.5 Expanded Granular Sludge Blanket (EGSB) Reactor

EGSB reactor is a modified version of the UASB reactor in the sense that a higher upflow velocity is achieved in the reactor by increasing the reactor height and/or effluent recycle (Lettinga et al., 1993). The granular sludge bed in the reactor is operated in the expanded mode as a result of higher upward velocity applied, that is 6-12 m/h against less than 1-2 m/h in a UASB reactor (van Haandel and Lettinga, 1994). The EGSB reactor has ben shown to be fairly efficient in the removal of soluble organic matter even at low temperature due to the intensive contact between the organic matter and the sludge granules. This system seems to be particularly useful at lower temperature and low-strength wastewaters, when the gas production rate

and consequently, the mixing intensity induced by it, are relatively low.

However, the system is reported to be inadequate for the removal of particulate organic matter due to the high upflow velocity in the reactor. The influent suspended solids are 'blown' through the granular bed and leave the reactor with the effluent (van Haandel and Lettinga, 1994).

### 2.6.6 Upflow Anaerobic Sludge Bed Filter (UBF) Reactor

During the last decade, many hybrid reactors have been developed, which are combinations of the basic reactor types (Tilche and Vieira, 1991; Boopathy and Tilche, 1991). In an upflow sludge bed filter, a filter type of packing media is provided in the settler zone of UASB (Guiot and van den Berg, 1984). The filter zone above the UASB was mainly expected to prevent the escape of suspended solids achieving high biomass concentration in the reactor and thus allowing the treatment of dilute and high strength wastewater. A low-strength (300 mg/L COD) wastewater could be treated with 95% efficiency at an HRT of 3 h using this type of reactor (Droste et al., 1987). However, in this study, serious sludge washout and deterioration of effluent quality was observed at 1.6 h HRT corresponding to a reactor upflow velocity of about 0.4 m/h. In these reactors, probably, the sludge particles were easily forced through the filter by the flow of liquid and gas especially at low HRTs.

## 2.7 Comparison of the Performance of Anaerobic Sewage Treatment Process

Van Haandel and Lettinga (1994) give a compilation of performance

data for several pilot and full-scale anaerobic treatment plants. For all treatment systems considered, a linear relation between the logarithms of removal efficiency and the applied retention time were shown to exist. This relationship can be written as

$$HRT = [(1 - E)/c_1]^{c_2} \qquad [2.24]$$

where HRT = Hydraulic retention time

E = Efficiency of organic material removal

$c_1$ and $c_2$ = constants, characteristic of the different anaerobic treatment processes.

The values of these constants are listed in Table 2.3 (van Haandel and Lettinga, 1994). The calculated values of the retention time required to achieve an organic material removal efficiency of 80% with the different systems are also given in the table. In the range of practical interest, the performance of a UASB reactor and a fluidised or expanded bed reactor tend to be similar with the same retention time. However, the last two systems have the important disadvantage of the need for additional pumping. Moreover, the fluidised bed does not seem to be adequate for sewage treatment because of the difficulties of retaining influent suspended solids in the reactor. Consequently, the UASB concept looks the most attractive option among these for sewage treatment (van Haandel and Lettinga, 1994).

Granulation becomes almost impossible in the case of full-scale UASB units treating low-strength wastewater like sewage. Though, experience from full-scale UASB reactors demonstrates that sludge

Table 2.3 Empirical values of the characteristic constants and HRT for 80% COD removal for different anaerobic system (temperature >20°C) (adapted from van Haandel and Lettinga, 1994)

| System | $c_1$ | $c_2$ | HRT for E = 0.8 (h) |
|---|---|---|---|
| UASB | 0.68 | 0.68 | 5.5 |
| Fluidised or expanded bed | 0.56 | 0.60 | 5.5 |
| Anaerobic filter | 0.87 | 0.5 | 20 |
| Anaerobic pond* | 2.4 | 0.5 | 144 (= 6 d) |

* BOD removal efficiency.

granulation is certainly not a prerequisite for successful treatment of sewage, presence of granular sludge in reactor can offer many specific advantages (van Haandel and Lettinga, 1994). Compared to the flocculant sludge, the very compact nature of granules helps in achieving a higher biomass content in the reactor. The flocculant sludge falls apart under condition of mild mixing, while granular sludge stay intact even under fairly extreme conditions of hydraulic stress (Hulshoff Pol, 1989). Another major attraction of granular sludge is its high specific waste stabilization capacity (Lettinga et al., 1983). Thiele et al. (1988) suggested an enhanced syntrophic relation in granules by the juxtapositioning of the obligate hydrogen producing acetogens and hydrogen consuming methanogens. Some of the recent studies on the microstructure of UASB granules treating complex wastewater suggest the development of syntrophic microcolonies of these two groups of organisms

around the acetoclastic *Methanothrix* central core (MacLeod et al., 1990; Fang et al., 1994). This symbiotic association of the members of the large consortium, reduces the diffusion distance of metabolites and thus minimises the mass transfer and threshold limitations (Pavlostathis and Giraldo-Gomez, 1991; Thiele et al., 1988; Gujer and Zehnder, 1983). Granulation can create a micro-environment protecting the cells from predators and other unfavorable growth conditions (Hulshoff Pol, 1989).

Granulation in reactors is greatly influenced by the organic loading rate. Hickey et al. (1991) suggested a minimum sludge loading rate of 0.3-0.5 g COD/g VSS.d and a space loading rate of 5 g COD/L.d to accelerate granulation. For a low-strength wastewater (< 500 mg/L COD), these loading rates demand HRTs less than 2.5 h. This will result in superficial velocities in the settlers of full-scale units, far in excess of the permissible value of 0.5 m/h for flocculant seed sludge during start-up (Lettinga and Hulshoff 1992) or 0.7 m/h during regular operation with flocculant sludge (Tilche and Vieira 1991). Thus, it may not be possible to retain the flocculant seed sludge in the reactor simultaneously achieving required organic loading rate, for low-strength wastewater.

Moreover, even if reactors are seeded with granular sludge it is difficult to maintain the required loading rate with low-strength wastewater, limiting the average superficial velocity within 1-1.25 m/h, as suggested by Lettinga and Hulshoff (1992) for granular UASB. The low organic loading rates cause floatation and easy washout of granules from

the reactor, possibly due to hollow core development in the granules (Kosaric and Blaszczyk, 1990).

Expanded granular sludge bed (EGSB) reactors are reported to perform better compared to the conventional UASB especially at low temperatures and relatively low-strength wastewaters where the gas production rate is low to effect sufficient bed mixing. However, the reactor is not capable of removing suspended organic matter due to the high upflow liquid velocity. Moreover, this configuration requires the installation of an additional pump for recirculation. Recirculation of the effluent will dilute the incoming wastewaters and will thus reduce the substrate concentration gradient in the biomass.

Upflow anaerobic sludge bed filter (UBF) reactor, a combination of UASB and AF, has the capacity to retain high biomass concentration even without granular sludge. In this configuration the trapped suspended solids are likely to accumulate in the filter zone itself as no provision is delibeıately made to put it back to the sludge zone. Thus the sludge particle are easily forced out of the reactor especially at low HRTs.

## 2.8 Conclusion

Anaerobic treatment of domestic wastewater would be an attractive solution for environmental protection, particularly for developing countries. Among the various high rate anaerobic reactors, the UASB concept looks to be the most attractive option for sewage treatment. However, the process suffers from certain limitations. Granulation is almost impossible in the case of full scale UASB units treating

low-strength domestic wastewaters. Availability of granular sludge in the reactor is beneficial for various reasons discussed earlier. For low-strength wastewaters and at low temperature, the gas production rates will not be sufficient to effect the required bed expansion and mixing and thus affects substrate utilisation rate. Many investigators have tried for modification or hybridisation of the basic UASB concept and were successful in minimising above limitations to some extent.

Literature on the UASB full-scale experience indicates that a considerable fraction of the effluent COD is constituted by suspended organic matter. Reactor treating domestic wastewater at 4-6 h HRT are reported to exhibit an average COD (total/total) removal efficiency of 65% against a COD (total/filtered) removal efficiency of 80% (Lettinga, 1992). The escape of organic solids with the effluents contribute significantly to the effluent COD and often effects violation of the standards prescribed for their disposal into inland waters. A separate post-treatment in the form of oxidation ponds usually had to be incorporated to produce effluents conforming to the standards. Thus, there is an urgent need to develop a reactor configuration to avoid the separate post-treatment and at the same time to produce effluents conforming to the standards.

The reactor configuration to be developed should have an effective gas-liquid-solid separation device (GLSS) which can minimize the escape of suspended organic matter from the effluent at extremely low HRTs. This should effect retention of flocculant type seed sludge at HRTs demanded by the loading rates favourable for granulation with

low-strength wastewater. The modified configuration should achieve bed expansion while treating low-strength waste without any effluent recycle. The trapped/settled biomass in the GLSS should slide back to the digestion zone to become, once again, part of the active sludge bed. With this idea, a reactor configuration was developed and designed by incorporating the concept of tube settlers to replace the sedimentation zone and GLSS of conventional UASB reactor.

# 3. SCOPE OF THE PRESENT INVESTIGATION

The upflow anaerobic sludge blanket (UASB) reactor being the most attractive alternative for domestic wastewater treatment, efforts directed towards minimisation of its limitations would be worthwhile. Accordingly, this research work was conducted along the following lines:

(1) Development, design and fabrication of a new reactor configuration by introducing tube settlers in the place of the settler zone of a conventional UASB reactor aiming the following:

  (a) Proper separation of the three phases namely, gas, liquid and solid (biomass) in the reactor.

  (b) Retention of the flocculant seed sludge in the reactor even at low HRTs that are required for achieving the organic loading rates conducive for granulation with low-strength wastewater.

  (c) Maximum capturing of the suspended organic particulates and thus decreasing the effluent COD due to suspended solids.

  (d) Return of the settled biomass to the digestion zone to become, once again, part of the active sludge bed.

(2) Development of granular sludge from flocculant seed sludge using a low-strength wastewater (COD - <500 mg/L) in the modified UASB reactor.

(3) Performance evaluation of this modified reactor configuration while treating low-strength synthetic wastewaters based on a completely soluble as well as a complex substrates at HRTs ranging from 1-5 h.

(4) Evaluation of the contribution of the digestion as well as the settler zones in achieving the total and soluble COD removal.

(5) Evaluation of the effect of inclination of tube settler on the performance of the reactor.

(6) Evaluation of the suitability of the reactor configuration to produce effluents conforming to standards while treating raw domestic wastewater.

# 4. THE REACTOR CONFIGURATION DEVELOPMENT AND DESIGN

A need was felt to develop a reactor configuration by modifying the gas-liquid-solid separator (GLSS) of a conventional UASB reactor with the aim of minimising the escape of suspended solids with effluent even at extremely low HRTs. Tube/plate settlers are known to be very efficient in achieving sedimentation of suspended particles at short HRTs. Culp et al. (1969) have reported about the effective usage of tube settlers for clarification of sewage in a contact stabilisation process. Reid (1971) have used an upflow parallel plate settler inclined at $60^{o}$ with horizontal in an extended aeration system. This plant has exhibited a BOD and suspended solids removal efficiency of 96%. In these cases, the settlers were designated only to effect proper sedimentation of suspended solids.

In the present research work, it was intended to incorporate tube settlers on top of the digestion zone of a UASB reactor to replace the conventional GLSS. An assembly of PVC tubes kept at desired inclination can be supported on polyethylene mesh with a proper opening size which permits easy passage of suspended solids. This arrangement of the tubes is expected to effect proper gas-liquid-solid separation. The effluent collection system can be provided on top of the settler zone.

Sludge particles lifted by the adhering gas bubbles along with the treated liquid will enter into the tubes through the mesh opening. As the biomass with the adhering gas bubble move against the top inside surface of the tubes, the frictional resistance offered by the tube

surface to the gas bubble is expected to effect the separation of gas bubbles from the biomass. The freed biomass can then settle in the tubes and slide down to the sludge zone through the mesh opening. Formation of biofilm may also be possible on the tube surface.

For tube settlers, the relationship between critical particle settling velocity and the average flow velocity (Yao, 1973) has the following form

$$\frac{V_{sc}}{V_o} (\sin\theta + L_t \cos\theta) = \frac{4}{3} \qquad [4.1]$$

where $V_{sc}$ = critical settling velocity ($LT^{-1}$); any suspended particles with its settling velocity greater than or equal to this settling velocity would be completely removed

$V_o$ = the average flow velocity through the tube ($LT^{-1}$)

$\theta$ = angle of inclination of the tube with horizontal

$L_t$ = the relative length of the tube equal to $l/d$

where $l$ = length of the tube (L)

d = diameter of the tube (L)

The relative length $L'_t$ for the initial region of turbulence where the flow gradually changes to laminar flow due to the influence of the solid boundary on a circular tube is given by the equation

$$L'_t = 0.058 \frac{V_o d}{\nu} \qquad [4.2]$$

where $\nu$ = the kinematic viscosity of the wastewater ($L^2T^{-1}$).

The design of the bench scale units were based on the following reasoning. Hickey et al. (1991) suggested a space loading rate of 5 g

COD/L.d and a sludge loading rate of 0.3-0.5 g COD/g VSS.d to accelerate biomass granulation. For a wastewater of 500 mg/L COD, the required space loading rate can be achieved at an HRT of 2.4 h. Assuming a reactor VSS concentration of 10 g VSS/L (Lettinga, 1992), a specific loading rate of 0.4 g COD/g VSS.d can be achieved at an HRT of 3 h. With a reactor height of 4 m, as suggested by Lettinga (1992) in full scale units treating domestic wastewater, the liquid upflow velocity will be 1.67 m/h.

Assuming that 90% of the reactor cross sectional area would be available for liquid flow in the settler, the average flow velocity in the tubes corresponding to the velocity of 1.67 m/h would be 3.1 cm/min. The bench-scale reactor used in this study was designed to retain the flocculant type of seed sludge in the reactor for a flow velocity of 3.1 cm/min in the tube settler.

PVC tubes of 2 cm $\phi$ and 54 cm length were provided. With kinematic viscosity of 0.8 X $10^{-2}$ $cm^2$/sec at a temperature of $30^{o}C$ (Peavy et al., 1986) and average flow velocity of 3.1 cm/min, $L_t'$ was calculated to be 0.75 (eqn. 4.2). For the tube length of 54 cm, the total relative length ($L_t$ + $L_t'$) would be 27, and thus the relative length, $L_t$, available for settling would be 26.25. Using eqn. (4.1), for a tube inclination of $60^{o}$, the critical settling velocity, $V_{sc}$, was calculated to be 0.3 cm/min. This corresponds to 0.18 m/h which is less than the permissible superficial velocity of 0.5 m/h for retention of flocculant seed sludge during start-up (Lettinga and Hulshoff, 1992).

Providing a total reactor length of 1.4 m and a liquid column length of 1.24 m, the average flow velocity of 3.1 cm/min in tube would correspond to an HRT of 0.74 h. Hence at this HRT, in the bench-scale unit with a settler inclination of $60^{o}$, not only that the seed sludge would not be washed out, but suspended solids with settling velocity ≥0.18 m/h also would be completely retained. For a reactor with settler inclination of $45^{o}$, the settling efficiency would be still better.

In a full-scale reactor, similar settling efficiency can be obtained at 2.4 h HRT if the same relative length of 26.25 is maintained. It can be seen that this is possible even with tubes of higher diameter without sacrificing the depth of sludge zone of a conventional UASB reactor. For a tube diameter of 5 cm and flow velocity of 3.1 cm/min, $L'_t$ is calculated as 1.9. For a relative length, $L_t$, of 26.25, the total length of the tube required is 141 cm. If the tube inclination is $60^{o}$, the settler height required will be 122cm. For a total reactor height of 4 m in full scale conventional UASB reactor, the height of settler may be taken as 1.5 m (Lettinga and Hulshoff, 1992; Tilche and Vieira, 1991). In this space, the tube settler can be conveniently accommodated.

Based on the design given earlier, two bench-scale units of the modified UASB reactors, $R_1$ and $R_2$ with settler inclination of $45^{o}$ and $60^{o}$ respectively with the horizontal, were fabricated (Figure 4.1). Reactors were made-up of moulded acrylic sheet and had a total length of 1.4 m each with a liquid column length of 1.24 m. With an internal diameter of approximately 10 cm, the empty bed liquid volume was 9.16

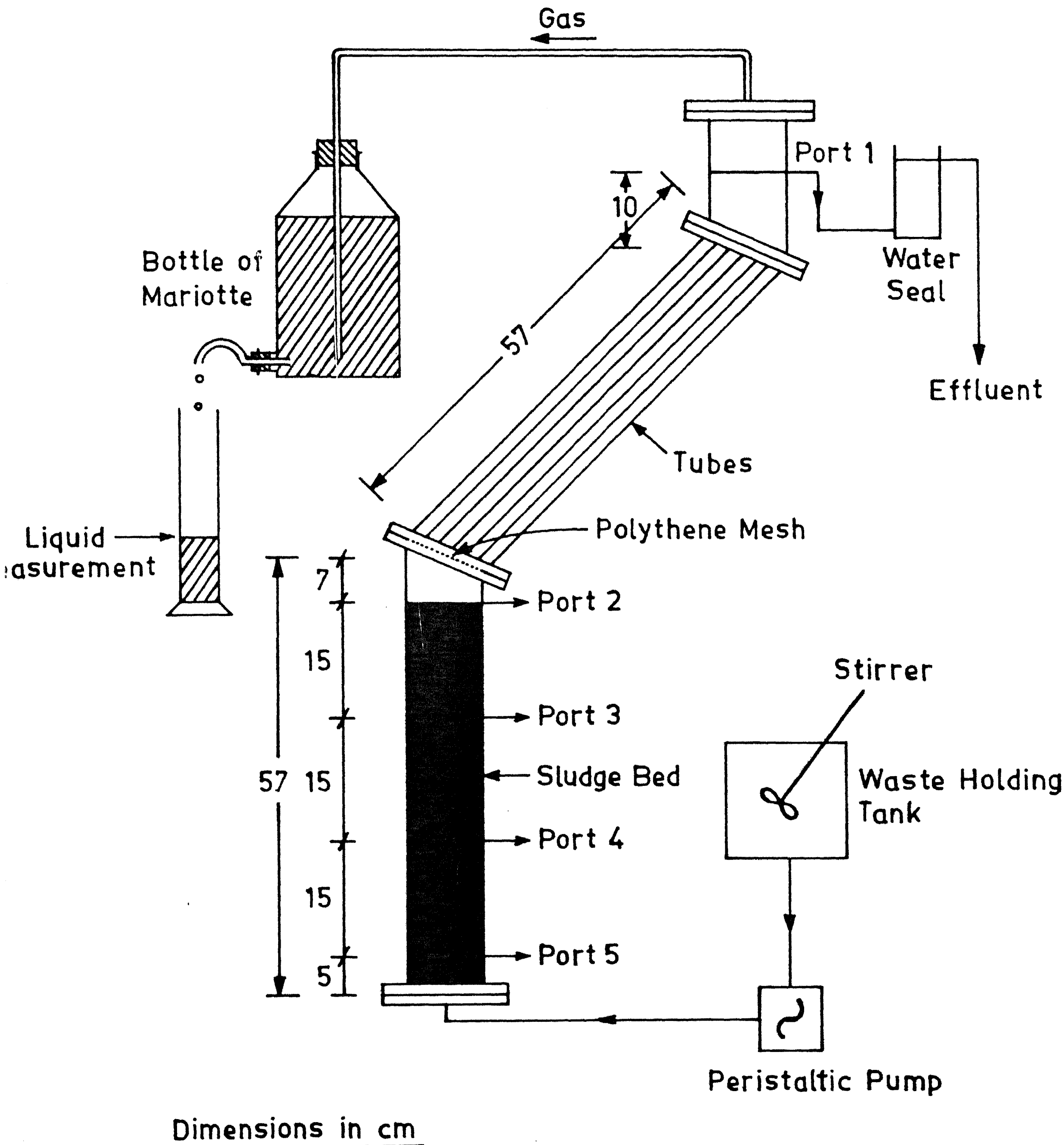



Fig. 4.1. Process Flow Diagram of the Modified UASB Reactor.

litre. The reactors had both the vertical sludge zone height and the settler zone length of 57 cm each. The inclined settler zone contained 19 PVC tubes of 2 cm diameter and 54 cm length supported on polyethylene mesh with 1.6 cm square openings. This settler zone was expected to act as the GLSS. Five sampling ports were provided at different heights (Figure 4.1) to facilitate withdrawal of samples from the reactor.

# 5. MATERIALS AND METHODS

Two bench-scale units of the modified UASB reactor were conceived, designed and fabricated as outlined in Chapter 4. These reactors were employed to treat three types of wastewaters at various HRTs. This chapter deals with the experimental set-up, characteristics of the wastewater and experimental methodology applied.

## 5.1 Materials

### 5.1.1 Experimental Set-up

Two units of the modified UASB reactors, $R_1$ and $R_2$ with the settlers inclined at angle of $45^o$ and $60^o$ respectively with the horizontal, were used in this study. Both the reactors had an empty bed liquid volume of 9.16 litres each and axial liquid length of 1.24 m. Other details of the reactors are given in Chapter 4. Figure 4.1 shows a schematic diagram of the experimental set-up. The wastewater was stored in a holding tank made of polyethylene, 110 litre capacity provided with a speed regulated stirrer (REMI, India) operated through a timer, stirring the wastewater for 6 minutes in every 30 minutes. The waste was pumped into the reactors using peristaltic pumps (PP 20, Miclins, India). The feed was distributed at the reactor bottom through a distributor containing five openings of 2-3 mm diameter. Sampling port Nos. 2 to 5 were used to withdraw sludge samples from the reactor. The treated effluent was collected from port No. 1 through a water seal. The gas production rate was measured with the help of a Mariotte bottle by liquid displacement of saturated sodium chloride solution containing

5% by volume $H_2SO_4$ and methyl orange. Both the reactors were operated at ambient temperature throughout this study.

### 5.1.2 Wastewaters

Three types of wastewaters were used in this investigation. The first two were synthetic wastewaters based on sucrose and a commercial grade babyfood (CERELAC, NESTLE India Ltd.). The third one was raw domestic wastewater collected from the campus of I.I.T. Kanpur.

#### 5.1.2.1 Synthetic Wastewater

The synthetic wastewaters were prepared in tap water using sugar or CERELAC and adding other growth nutrients as outlined in Table 5.1. Required amount of bicarbonate was also added to maintain the reactor pH in the range of 7-7.3.

Table 5.1: Synthetic nutrient media composition* (Kroeker et al., 1979)

| Compound | Concentration, mg/L |
|---|---|
| $KH_2PO_4$ | 4000 |
| $MgSO_4.7H_2O$ | 126 |
| $CoCl_2.6H_2O$ | 36 |
| $FeCl_3.6H_2O$ | 864 |
| $CaCl_2.6H_2O$ | 600 |
| Urea | 4000 |
| Yeast extract | 400 |

* The medium constituted one by twenty-fifth of the feed.

The average COD and BOD of the sucrose wastewater during the entire period of the study were 485 mg/L and 466 mg/L respectively.

Table 5.2 gives the approximate composition of CERELAC used to prepare the wastewater. The COD, nitrogen and phosphorus content of CERELAC were determined and are presented in Table 5.2.

Table 5.3 gives the average characteristics of the CERELAC based wastewater.

Table 5.2: Approximate composition of CERELAC

| Constituent - 1* | g/100 g of product |
|---|---|
| Fat | 9 |
| Linoleate | 1.5 |
| Protein | 15.5 |
| Carbohydrates | 68.9 |
| Dietary fibre | 1.4 |
| Ash | 2.7 |
| Moisture | 2.5 |
| **Constituent - 2**** | **g/100 g of product** |
| COD | 137 |
| Total nitrogen-N | 1.4 |
| Total phosphorus-P | 0.54 |

* as per NESTLE, India Ltd. ** as per analysis

Table 5.3 Average composition of the synthetic CERELAC wastewater

| Constituent | Concentration, mg/L |
|---|---|
| Total COD | 476 |
| Soluble COD | 205 |
| Total $BOD_5$, $20^oC$ | 322 |
| Soluble $BOD_5$, $20^oC$ | 163 |
| VSS | 94 |
| TSS | 115 |

#### 5.1.2.2 Raw Domestic Wastewater

Domestic wastewater was collected from Sump Well No. 2 located in the residential area of Indian Institute of Technology, Kanpur (India). Before transferring to the waste holding tank, it was strained through a strainer of 1.5 X 2 mm size. The average characteristics of the wastewater used during the study is given in Table 5.4.

### 5.1.3 Seed Sludge

Sludge collected from the 5 MLD UASB plant treating domestic wastewater of Kanpur (India) was used to seed both the reactors $R_1$ and $R_2$. This seed sludge was found to be flocculent in nature on visual observation. The TSS and VSS contents of the seed sludge were 86 g/L

and 31 g/L respectively with a specific methanogenic activity (SMA) of 0.05 g $CH_4$-COD/g VSS.d at $30^oC$.

Table 5.4 Average composition of the Domestic Wastewater

| Constituent | Concentration, mg/L (except pH) | | |
|---|---|---|---|
| | Average | Maximum | Minimum |
| COD (total) | 280 | 622 | 125 |
| COD (soluble) | 74 | 200 | 43 |
| $BOD_5$, $20^oC$ (total) | 82 | 175 | 38 |
| $BOD_5$, $20^oC$ (soluble) | 30 | 80 | 16 |
| TSS | 660 | 1575 | 171 |
| VSS | 208 | 550 | 76 |
| $NH_3$-N | 17 | 25 | 12 |
| Total nitrogen-N | 22 | 32 | 17 |
| Total phosphorus-P | 1.2 | 1.6 | 1.7 |
| $SO_4^=$ | 85 | 100 | 75 |
| pH | 7.8 | 8.3 | 7.1 |
| Alkalinity-$CaCO_3$ | 365 | 460 | 180 |

## 5.2 Methods

### 5.2.1 Schedule of Reactor Operation

During the first phase of the study, the reactors were subjected to an initial "primary start-up" operation using the synthetic

sucrose wastewater. This process, according to Lettinga and van Haandel (1993), is the cultivation of a sufficient amount of well-adapted anaerobic sludge from a poor-quality seed sludge. The guidelines prescribed by Hulshoff Pol (1989) for start-up of UASB reactors were followed, in general, during the "primary start-up" and granular sludge development. Required amount of the seed sludge was added to both the reactors to get a reactor VSS concentration of 6 g/L. The reactors were started initially at 12 h HRT on sucrose wastewater. The space loading rate was gradually increased by reducing the HRT, while keeping the wastewater strength constant to achieve loading conditions conducive for granulation. Performance of both the reactors were evaluated after the attainment of pseudo-steady-state (PSS) conditions at 5, 4, 3, 2.4 and 2 h HRTs by conducting detailed studies on the liquid, solid (biomass) and gas phases of the reactors. This phase of the study with sucrose wastewater which included primary start-up, granular sludge development and performance evaluation at various HRTs lasted for about 220 days.

The next phase of the study was designed to evaluate the suitability of the modified UASB reactor configuration for treatment of wastewater containing complex organic substrate. For this purpose a synthetic wastewater based on CERELAC was used and the already developed granular sludge was adapted to this wastewater during a "secondary start-up" operation. Thereafter, reactor $R_1$ was operated at HRTs of 4 and 2 h and $R_2$ was operated at 4, 2 and 1 h keeping the wastewater strength constant to get PSS conditions. The total period of reactor operation during this phase of the study was about 115 days.

In the final phase of the study, the suitability of the reactor configuration to treat raw domestic wastewater at low HRTs was evaluated. The HRT of reactors $R_1$ and $R_2$ containing granular sludge was increased to 3 h. During the initial 8 days of "secondary start-up", the feed was gradually changed from CERELAC-based wastewater to domestic wastewater. Thereafter $R_1$ and $R_2$ were operated at 3 h and 2 h HRT respectively to get PSS condition. The reactor $R_1$ alone was further operated at 1 h HRT.

After collection of 5 days PSS data, $R_1$ and $R_2$ were operated without the tubes in the settler zone at HRTs 1 h and 2 h respectively to evaluate the role of tube settlers in the reactor performance. The entire third phase of study had lasted for about 125 days.

All throughout the study with the three types of wastewaters, the reactors were operated at ambient temperature. Excess sludge produced in the reactors was withdrawn almost every day from port No. 2, maintaining a constant sludge bed height of 50 cm.

### 5.2.2 Schedule of Analysis

The reactor performance was evaluated based on the composited influent and effluent samples during this study. The waste holding tank was filled at 12 h interval with either freshly prepared synthetic or the raw domestic wastewater. Time weighted samples of wastewater from the influent tank taken just after and prior to filling was preserved under refrigeration. These samples collected over a day was used for analysis. The effluent samples collected over a day and

kept under acidified condition was used to determine the effluent parameters.

The reactor temperature, pH and gas production rate were monitored daily throughout the study. COD of influent and effluent, total alkalinity and VFA of the effluent and the reactor sludge bed height were determined on alternate days during synthetic wastewater feeding. During PSS at each HRT, along with the above mentioned parameters, gas composition, effluent VSS and TSS and COD of settled sample from port No. 2 were determined daily for 5 consecutive days and the average values were calculated. When the reactors were receiving domestic wastewater, pH, COD, total alkalinity, VFA, VSS and TSS of influent and effluent were analysed on alternate days. During PSS, all the above parameters were determined daily for 5 days and the average values were calculated.

Influent and effluent $BOD_5$ were analysed on two days during a steady-state and the computed $BOD_5/COD$ ratio was used to project the PSS average $BOD_5$ value for all three types of wastewater. The excess sludge withdrawn everyday, from port No. 2, was preserved under refrigeration and the sludge collected in five days were analysed to determine the VSS and TSS contents. The value of total VSS and TSS withdrawn in five days were divided by the total litres of waste treated during 5 days to get the PSS sludge wasting. The reactor performance along the height was evaluated by drawing samples from the bottom four sampling ports on the last day of each PSS condition. The specific methanogenic activity

(SMA) of the individual sludge samples drawn from different ports were averaged to get the reactor SMA.

### 5.2.3 Analytical Techniques

#### 5.2.3.1 COD and BOD

The COD and BOD of samples were determined as per the methods outlined in Standard Methods (1989).

#### 5.2.3.2 Volatile Fatty Acids (VFA) and Total Alkalinity (TA)

Direct titration method described by DeLallo and Albertson (1961) was used to determine the VFA and TA. This involves titration of the samples with strong acid to pH 4.3 which gives alkalinity due to bicarbonate, VFA and phosphates. When the sample pH is reduced to 3.3, bicarbonate ions will be converted to carbonic acid and subsequent boiling of the sample removes all of the carbonic acid as carbon dioxide. The back titration from pH 4 to 7 measures the alkalinity due to organic acids. The conversion factor for determination of VFA from volatile acids alkalinity depends on the proportion of acid which is titrated between pH 4 to 7.

A 25 mL of centrifuged sample was titrated to a pH of 4.3 with 0.1 N sulphuric acid. The volume of acid used was noted and titrated was continued to pH of 3.5-3.3. The sample was gently boiled for three minutes and cooled in a water bath to room temperature. It was then titrated against 0.05 N NaOH upto pH 4. After noting the burette reading, the titration was continued to pH 7 and final reading was noted. VFA concentration was calculated as follows:

VFA Alkalinity, mg/L as $CaCO_3$ = (mL 0.05 N NaOH $x$ 2500)/mL sample

VFA, mg/L as $CH_3COOH$ = VFA Alkalinity $x$ 1.5

The multiplying factor 1.5 takes care of the conversion of $CaCO_3$ alkalinity to VFA expressed in terms of $CH_3COOH$ and the assumption that 80% of VFA is titrated from pH 4 to 7.

Total Alkalinity, mg/L as $CaCO_3$ =

$$\frac{\text{mL 0.1 N } H_2SO_4 \text{ (used upto 4.3) } x \text{ 5000}}{\text{Volume of sample (mL)}}$$

**5.2.3.3 TSS and VSS**

The TSS and VSS of the various samples were determined as per the Standard Methods (1989).

**5.2.3.4 Sulphates**

The sulphate present in the domestic wastewater was determined by the turbidimetric method as given in Standard Methods (1989).

**5.2.3.5 Ammonia Nitrogen and Total Nitrogen**

Ammonia nitrogen present in domestic wastewater was determined by Nesslerisation method as per Standard Methods (1989).

Total nitrogen was determined by the method given by Thompson and Morrison (1951). In a 100 mL Kjeldal flask, 10 mL of the sample (containing 5-200 $\mu$g of nitrogen) was taken to which 4 mL of 3 N sulfuric acid was added. Glass beeds were added to avoid bumping, and digestion was carried out for 10 minutes after the white fumes started appearing. After cooling, contents of the Kjeldal flask were transferred to a 50 mL beaker and the pH was adjusted to about 7.0 with

1.25 N NaOH. Total volume was made upto 25 mL in a standard volume flask and 10 mL or aliquot diluted to 10 mL was taken for Nesslerisation. Nesslerisation was done as given in Standard Methods (1989).

**5.2.3.6 Total Phosphorus**

The total phosphorus present in the CERELAC and domestic wastewater was determined as per the Standard Methods (1989).

**5.2.3.7 Gas Compositions Analysis**

The composition of biogas was analyzed using a gas chromatograph (NUCON-5700, India) equipped with a carbosphere column of 3 mm diameter and 1.83 m length and a thermal conductivity detector (TCD). The injector, oven and detector temperature were maintained at 70°C, 60°C and 70°C respectively. Hydrogen, at a flow rate of 30 mL/min was used as the carrier gas. The bridge current for the TCD was 100 mA.

**5.2.3.8 Specific Methanogenic Activity (SMA)**

A procedure outlined by Valcke and Verstraete (1983) with certain modification was used to determine the specific rate of acetoclastic methanogenesis of the sludge samples. This procedure is based on the findings that during a maximum incubation period of 24 h, biomass growth is minimal and acetate conversion rate obeys zero order kinetics and is not affected by substrate concentrations between certain limits (Lawrence and McCarty, 1969).

The sludge sample was first diluted to get a VSS concentration of approximately 5 g/L with a mineral solution of composition as given in Table 5.5.

Table 5.5: Composition of mineral solution for determination of SMA*

| Compound | Concentration, mg/L |
|---|---|
| $KH_2PO_4$ | 2500 |
| $K_2HPO_4$ | 1000 |
| $NH_4Cl$ | 1000 |
| $MgCl_2$ | 100 |
| Yeast extract | 200 |
| $Na_2S.7H_2O$ | 100 |

* Valcke and Verstraete (1983)

To a series of 500 mL flasks, 350 mL each of this diluted sludge was added. The samples were subsequently acclimated for 24-48 h at 30°C without the addition of substrate. Thereafter, increasing amounts of sodium acetate as substrate was added to get sludge loading in the range of 0.3 to 1.0 g acetate/g VSS. Care was taken to introduce as little oxygen as possible into the liquor. The pH of sludge samples was adjusted to 6.7 either with 1 N HCl or 1 N NaOH. Then the flasks were flushed with nitrogen gas for at least 1 minute and incubated at 30°C for 24 h in a water bath shaker (Narang Scientific Works, India) to which an additional arrangement to activate the shaker every 6 minutes in half an hour was incorporated.

The gas produced was bubbled through 1 N NaOH solution to remove $CO_2$ and the volume of $CH_4$ produced was measured by liquid displacement.

Gas measurements were noted upto 24-30 h, after which the VSS content of the sludge sample was determined. The maximum specific methane production rate in mL $CH_4$/g VSS.d was calculated. If the maximum $CH_4$ production had occurred at the highest sludge load, higher sludge loads than 1.0 g acetate/g VSS were employed. Considering the theoretical COD of methane (equal to 4 g COD/g $CH_4$) the maximum specific methane production rate was expressed in terms of g $CH_4$-COD/g VSS.d and was reported as the specific methanogenic activity (SMA) of the sludge.

# 6. RESULTS AND DISCUSSION

## 6.1 Introduction

In the present investigation, two units of the modified UASB reactor (Fig. 4.1) with empty bed volume of 9.16 liters each were employed. Both the reactors ($R_1$ and $R_2$), with the settler compartment inclined at $45^o$ and $60^o$ respectively with the horizontal, had an axial length of 1.24 m.

These reactors were used to develop granular sludge from flocculant type of seed sludge, using low strength soluble wastewater. Further, studies were conducted to evaluate the suitability of the reactor configuration to produce effluents conforming to standards at ambient temperature. Synthetic wastewaters, based on completely soluble as well as complex substrates, and raw domestic wastewater were used in the study.

In the first phase of the study, sufficient amount of active anaerobic sludge was developed by feeding synthetic wastewater with average COD of 485 mg/L containing sucrose as carbon source. The space loading was gradually increased by reducing the hydraulic retention time (HRT) to achieve loading rates conducive for granulation. Reactor performance was evaluated after attaining pseudo-steady-state (PSS) condition at HRTs varying from 5 to 2 h, by conducting detailed studies on liquid, solid and gas phases of the reactor.

In the next phase of the study, the granular sludge developed was adapted to complex synthetic wastewater prepared from a commercial grade

baby food containing insoluble carbohydrate, protein and fat (CERELAC, NESTLE INDIA Ltd.). The reactor performance was studied at various HRTs ranging from 4 to 1 h with this wastewater.

In the final phase, the reactors were fed with domestic wastewater collected from a sump well in the residential area of Indian Institute of Technology Kanpur (India). The reactor performance was evaluated after achieving PSS condition at HRTs in the range of 3 to 1 h. The detailed results and discussion of these studies are presented in the following sections.

## 6.2 Operation of the Reactors during Primary Start-up and Development of Granular Sludge

The guidelines prescribed by Hulshoff Pol (1989) for start-up of UASB reactor was followed, in general, in this investigation during the primary start-up and granular sludge development stage. The reactors were seeded with sludge from the 5 MLD UASB plant treating domestic wastewater of Kanpur (India). The seed sludge was found to be flocculant in nature on visual observation after dilution. The TSS and VSS contents of the seed sludge were 86 g/L and 31 g/L respectively, and had a specific methanogenic activity of 0.05 g $CH_4$-COD/g VSS.d at 30$^{o}$C. Required amount of this sludge was added to both the reactors ($R_1$ and $R_2$) to get a reactor VSS concentration of 6 g/L. The reactors were started initially at 12 h HRT, feeding synthetic wastewater containing sucrose and other nutrients, as outlined in Table 5.1. Required quantity of bicarbonate was added to maintain the reactor pH in the range of 7.0-7.3.

The first phase of the study with sucrose wastewater lasted for about 220 days of continuous operation of the reactor. The data obtained on important process parameters for the initial 101 days of reactor operation which included primary start-up, granular sludge development and PSS at 5 h and 4 h HRTs are presented in Figures 6.1-6.4. The average COD and $BOD_5$ of the wastewater were 485 and 466 mg/L during these days.

### 6.2.1 Performance of Reactors

During the first 12 days of operation, the space loading rate was gradually increased from 0.96 to 2.32 g COD/L.d by reducing the HRT from 12 to 5 h. The corresponding increase in sludge loading rate, assuming that there was no significant change in the reactor sludge content, was from 0.16 to 0.38 g COD/g.VSS d. On day 14, soluble COD removal efficiency of 53% and 46% were observed in $R_1$ and $R_2$ respectively. The effluent VFA concentrations were 95 and 100 mg/L as $CH_3COOH$ and the reactor pH were 7.0 and 7.18 for $R_1$ and $R_2$ respectively. The maximum VFA level measured during this period was 130 mg/L.

Hulshoff Pol (1989) suggests that the acetate concentration in the reactor should be kept below 200 mg/L to promote the selective growth of *Methanothrix* over *Methanosarcina* as *Methanothrix* granules grow 4 to 6 times bigger than unstable *Methanosarcina* granules and are therefore more easily retained in the reactor.

As the VFA levels in both the reactors were well below the limit of 200 mg/L, the loading was increased by reducing the HRT to 4 h on day 14, though the COD removal efficiencies were not high (53% and 46%).

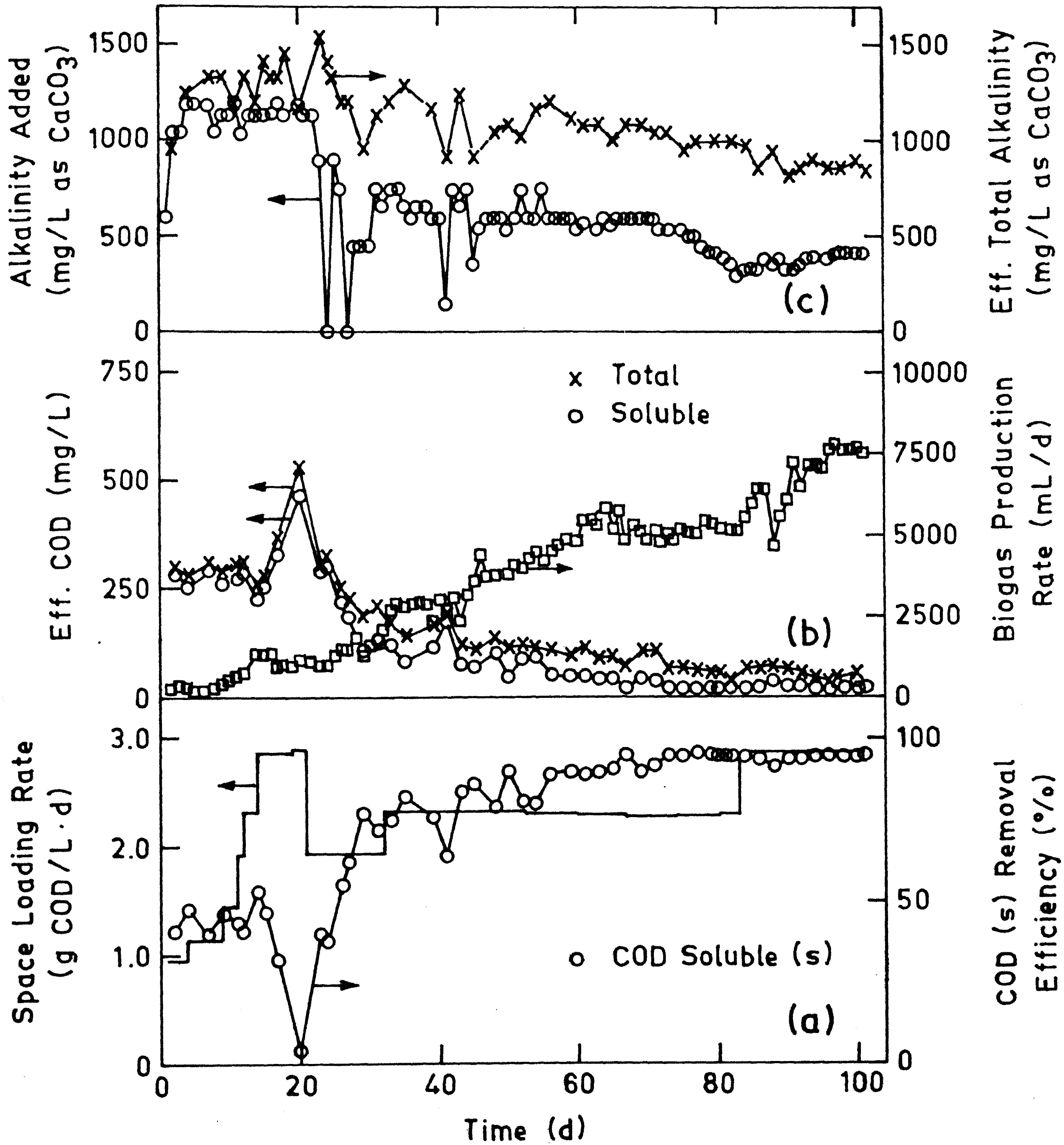



Fig. 6.1. Performance of $R_1$ Fed with Sucrose-Based Wastewater During Primary Start-up and Granular Sludge Development.

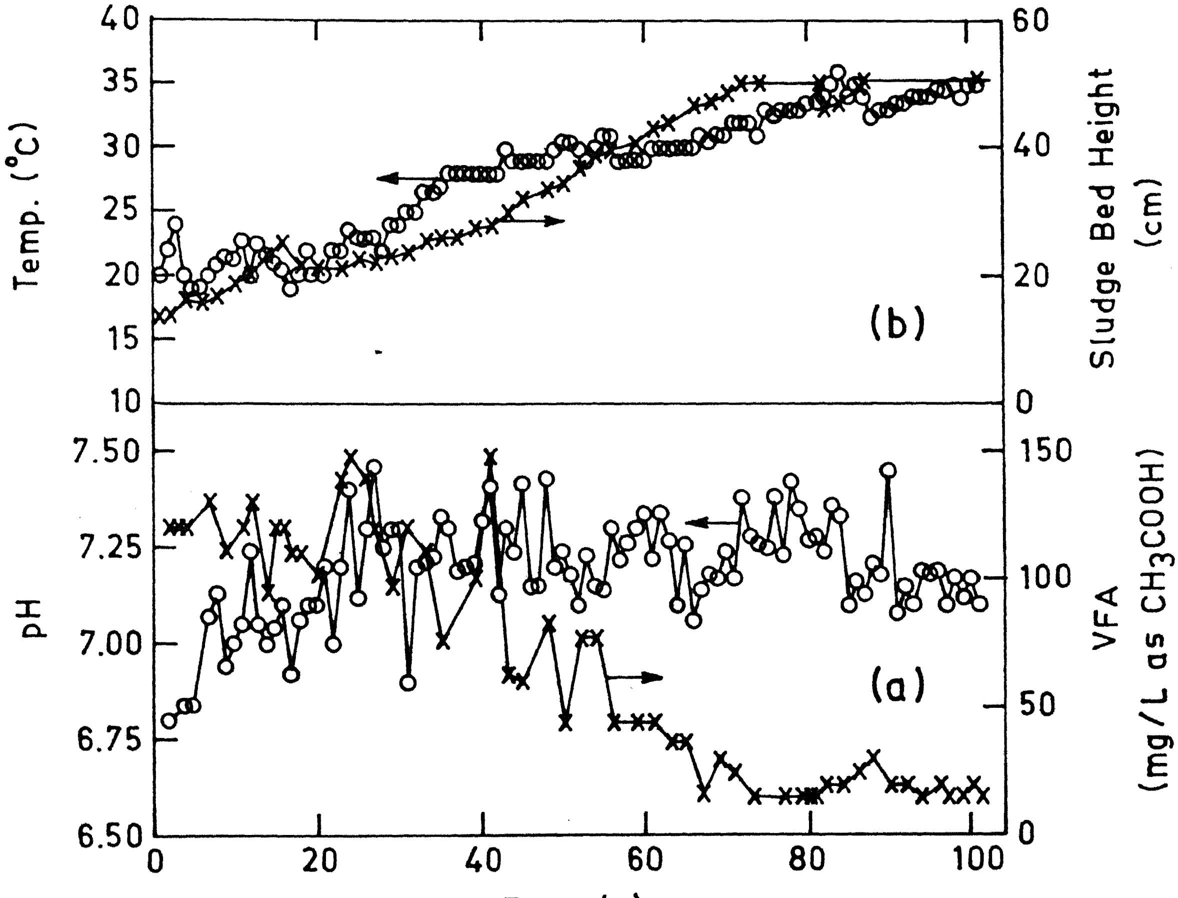



Fig. 6.2. Performance of $R_1$ Fed with Sucrose-Based Wastewater During Primary Start-up and Granular Sludge Development.

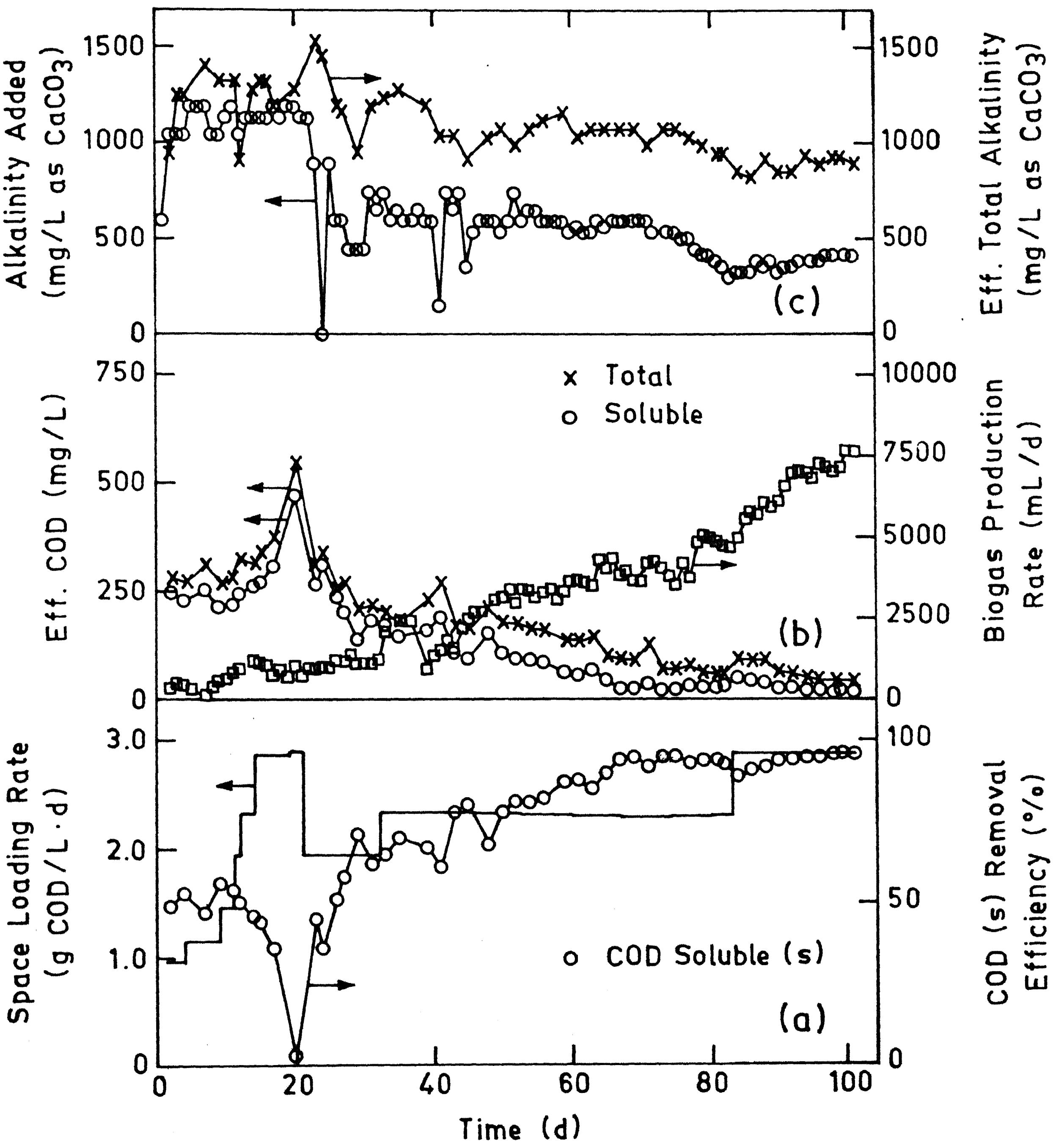



Fig. 6.3. Performance of $R_2$ Fed with Sucrose-Based Wastewater During Primary Start-up and Granular Sludge Development.

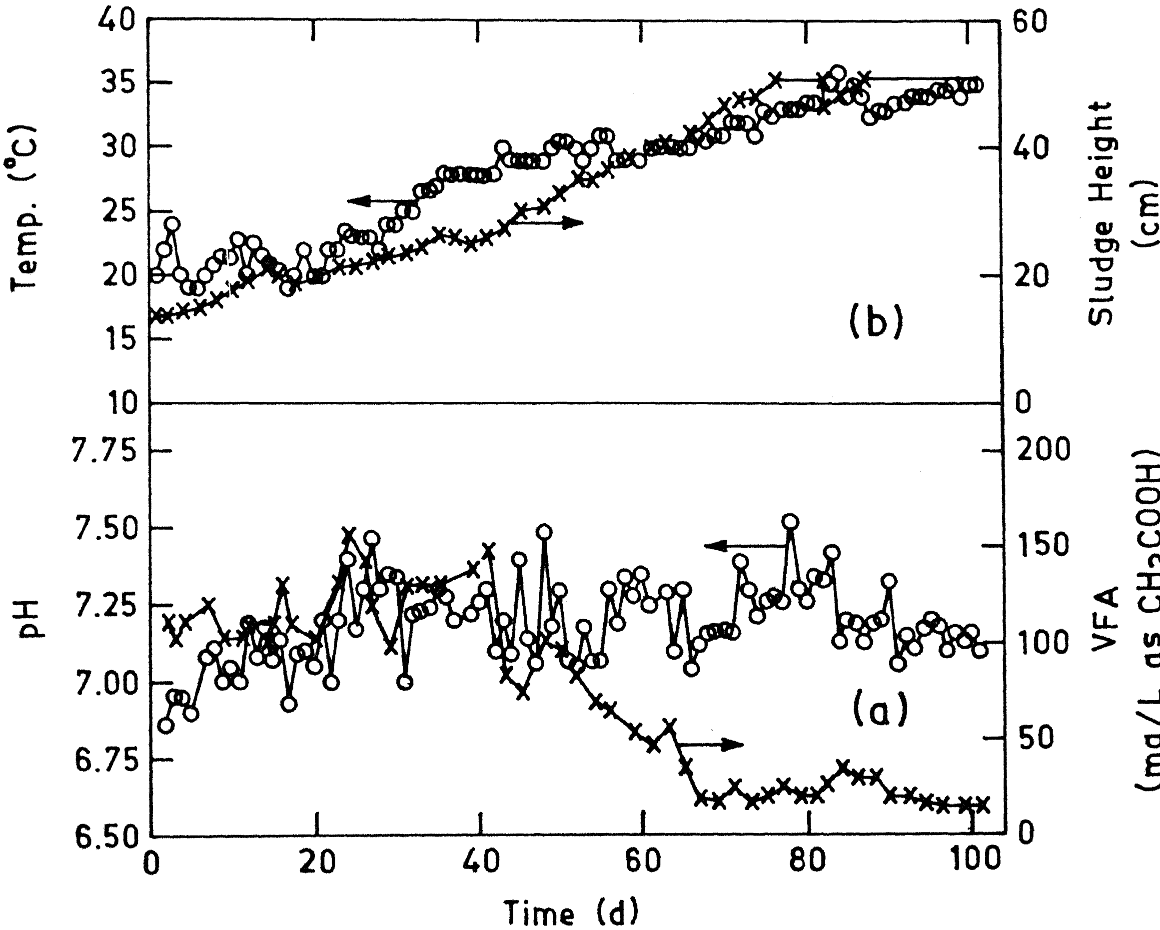



Fig. 6.4. Performance of $R_2$ Fed with Sucrose-Based Wastewater During Primary Start-up and Granular Sludge Development.

This resulted in a space loading of 2.88 g COD/L.d and sludge loading of 0.48 g COD/g VSS.d.

Following this, the effluent soluble COD of both the reactors $R_1$ and $R_2$ increased gradually reaching a maximum value of 465 and 470 mg/L respectively on day 20. The corresponding COD removal efficiencies were only 4% and 3%. This indicated that the loading rate was far beyond the specific substrate utilization rate of the sludge. The HRT was hence increased to 6 h on day 21 and the resulting loading rate of 1.93 g COD/L.d was maintained for the following 10 days. This reduction in space loading resulted in a gradual improvement in the effluent quality. The increase in reactor temperature from 20 to 25$^{o}$C might also have contributed to this. During these 10 days the maximum COD removal efficiencies in $R_1$ and $R_2$ were 77% and 71% respectively.

The HRT of both the reactors were reduced to 5 h on day 32, achieving a space loading rate of 2.33 g COD/L.d and was maintained till day 83. The soluble COD removal efficiency marginally improved and remained above 80% after day 48 for $R_1$ and after day 51 for $R_2$. The biogas production rate on these days was 180 and 152 mL/g COD applied for $R_1$ and $R_2$ respectively with 80% of methane content. From day 32 to 51 the temperature gradually increased from 25 to 30$^{o}$C.

### 6.2.2 Monitoring the Primary Start-Up

The amount of VSS present in the reactors was calculated from sludge VSS profile assessed from the samples drawn from sampling ports along the height of the reactors on day 52. Reactors $R_1$ and $R_2$ had a VSS concentration of 8.14g/L and 7.53 g/L respectively.

Corresponding to 80% soluble COD removal efficiency, the specific substrate utilization rates were 0.23 and 0.25 g COD/g VSS.d. The specific methane recovery rate (SMRR) for $R_1$ and $R_2$ were observed to be 0.12 and 0.11 g $CH_4$-COD/g VSS.d respectively. According to Lettinga et al. (1993) the specific methanogenic activity (SMA) exhibited by the sludge, once the start-up is complete, is about 0.1 g $CH_4$-COD/g VSS.d at $30^oC$. Comparing this SMA with the SMRR observed and the significant specific substrate utilization rate, it is evident that the sludge in both the reactors were active. During this period the alkalinity required to be added with the feed to maintain the reactor pH in the range of 7.0-7.3 was half of that required at the initial stage. This also indicated considerable growth of acetoclastic methanogens in the reactor. Thus, it is clear that the primary start-up of both the reactors had been completed by day 51.

#### 6.2.2.1 Comparative Performance of $R_1$ and $R_2$ during Primary Start-up

Even though both the reactor were seeded and operated in the same manner, $R_1$ had a marginally higher VSS content on day 52 than $R_2$ as mentioned earlier. The sludge heights in the reactors also indicated a more biomass accumulation in $R_1$ compared to $R_2$ (Figs. 6.2b and 6.4b). In general, during the period of primary start-up, $R_1$ had shown better soluble COD removal efficiency (Figs. 6.1a and 6.3a). Moreover, the effluent from $R_2$ contained more suspended solids COD than that from $R_1$. These observations indicate that reactor $R_1$, with the

settler inclined at $45^{o}$, has a better biomass retention capacity than $R_2$ during the initial start-up operation.

### 6.2.3 Granular Sludge Development

On continued operation of the reactors with the same space loading of 2.33 g COD/L d, biomass accumulated in both the reactors as evidenced by the gradual increase in sludge bed height (Fig. 6.2b and 6.4b). The sludge level reached to a height of 50 cm on day 72 in $R_1$ and on day 76 in $R_2$. Accordingly, the soluble COD removal efficiency also improved and reached 95% in both the reactors. Thereafter, leaving a clear space of 7 cm height below the settler zone, the excess sludge accumulated was withdrawn from port No. 2 almost every day. The sludge was preserved under refrigeration and the VSS and TSS of the total sludge wasted in five days were determined.

Steady state for 5 h HRT was assumed from day 78 to day 82 as the effluent COD and gas production remained fairly constant. Sludge samples withdrawn from the reactors during this period had not shown any visual evidence of granulation. Details of studies conducted to evaluate the reactor performance at the steady state are presented later.

On day 83 the space loading was increased to 2.88 g COD/L.d by reducing the HRT from 5 to 4 h and was maintained till day 101. After this load increase there was a transient shift in the effluent COD (Fig. 6.1a and 6.3a). However, the soluble COD removal efficiency reached 94% on day 90 for both the reactors. The reactor temperature was in the range of 34-35$^{o}$C during this period. Steady state was assumed during

the last five days and detailed studies were conducted to evaluate the reactor performance at this loading and the results are presented later.

Visual examination of sludge samples drawn from both the reactors indicated the presence of many granules of 1-3 mm $\phi$. The imposed space loading and sludge loading rates at this stage were 2.88 g COD/L.d and 0.24 g COD/g VSS.d respectively, for both the reactors. The gas production rate was 0.95 and 0.91 L/L.d in $R_1$ and $R_2$ respectively. Figure 6.5 is the photograph of the diluted sludge sample drawn from Port No. 4 at 4 h HRT, showing the granules.

In the present investigation granulation was observed at lower loading rate compared to the sludge loading rate of 0.3-0.5 g COD/g VSS.d and space loading rate of 5 g COD/L.d suggested by Hickey et al. (1991). Hulshoff Pol (1989) observed granulation in conventional UASB reactors, fed with synthetic wastewater of COD 3000 mg/L containing 95% sucrose and 5% VFA mixture, at a sludge loading rate of 0.3 g COD/g VSS.d. The imposed space loading was 4 g COD/L d. Granulation process was reported to proceed faster on carbohydrate substrate. Main reasons attributed to this were the higher growth yield, presence of other microflora in the sludge and higher extracellular polymer (ECP) production on carbohydrate substrate compared to VFA substrate.

A homogeneous and gentle agitation of the sludge caused by gas production also has an important role in the granulation process (Lettinga *et al.*, 1980, Lettinga and van Haandel 1993). At higher loading rates this natural mixing is rarely impaired. However, the gas production rate for a particular COD loading rate varies with the type


Figure 6.5 Photograph of diluted sludge sample from port No. 4 of $R_1$ fed with sucrose-based wastewater at 4 h HRT.

of wastewater and COD removal rate and this variation becomes critical in reactors operating at low loading rates. Manjunath *et al.* (1990) observed granulation at a space loading rate and sludge loading rate above 5 g COD/L.d and 0.5 g COD/g VSS.d respectively while treating cane sugar molasses based wastewater. When the reactors were operated at a space loading rate of 0.5-2.5 g COD/L.d and sludge loading rate of 0.1-0.3 g COD/g VSS.d, no granulation was observed. Reasons attributed for this was inadequate mixing of the digester content due to low gas production (0.1-0.5 L/L.d). In the granulation experiments of Hulshoff Pol (1989), a mechanical stirrer was used to mix the digester content intermittently. Thus the methane production rate effecting the required mixing, become critical, for the production of granular sludge.

In the present study using synthetic wastewater with sucrose as the sole carbon source, the gas production rate was about 0.9 L/L.d at a space loading rate of 2.88 g COD /L.d and a sludge loading rate of 0.24 g COD/g VSS d. This gas production rate which is based on total reactor volume, corresponds to 2.23 L/L.d based on the sludge volume. The above conditions might have brought about sufficient bacterial growth and sludge mixing resulting in granulation. It may be inferred that granulation is possible at relatively low loading rates if sufficient gas production and mixing thereby is ensured.

It is to be mentioned that during the 101 days of operation, at no time remarkable sludge wash out with the effluent was noted (Fig. 6.2b and 6.4b) The tube settler type of GLSS might have played a dominant role in holding the sludge during the start-up operation. However, once

the sludge had reached to a height of 50 cm, almost every day the excess sludge was being withdrawn from port No.2. This controlled sludge wasting from the top of the sludge bed, has helped in eliminating the light and finely dispersed biomass from the reactor and thus meeting one of the basic requirements for granular sludge development (Lettinga and van Haandel 1993). The higher biomass retention due to the controlled sludge wasting might have contributed to the high treatment efficiency, sufficient gas production and subsequent granulation.

### 6.3 Reactor Performance During Steady-state Operation With Sucrose-Based Synthetic Wastewater

Following the primary start-up, pseudo-steady-state operations of the reactors were carried out over a range of loading rates for a period of about 170 days. The loading rates were varied by changing the HRT but keeping the influent COD of the wastewater constant. During steady-state corresponding to each HRT, the reactor performance was evaluated for 5 consecutive days. Gas production rate and its composition, reactor pH, temperature, COD of influent, effluent and settled sample from port No. 2 and the effluent VSS and TSS were monitored daily. The sludge withdrawn from port No. 2 every day maintaining a sludge bed height of 50 cm, was preserved under refrigeration and was analyzed for VSS and TSS at the end. The reactor performance along the height was evaluated by drawing samples from the bottom 4 sampling ports on last day. The SMA of the individual sludge samples drawn from different ports were averaged to get the reactor SMA. Influent and effluent $BOD_5$ was analyzed on two days during a

steady-state and the obtained $BOD_5$/COD ratio was used to calculate the average $BOD_5$. Performance of $R_1$ and $R_2$ over the whole range of loading rate with sucrose-based wastewater is summarized in Table 6.1 and 6.2.

### 6.3.1 COD and BOD Removal Efficiency

Figure 6.6 shows the total and soluble COD removal efficiencies of reactors at various loading rates based on effluent samples from port No. 1 and settled samples from port No. 2. The soluble COD removal efficiency based on effluent samples was in the range of 94-95% for the loading range of 2.33-4.85 g COD/L.d (HRT 5-2.4 h) and decreased to 91% at 2 h HRT in both the reactors. The total COD removal efficiencies were in the range of 88-90% and 86-90% for $R_1$ and $R_2$ respectively up to 2.4 h HRT. This reduced to 84 and 83% at HRT 2 h. The soluble COD removal efficiencies based on samples from port No. 2 were in the range of 92-95% up to 2.4 h HRT and about 87% at 2 h HRT. This indicated that the biological activity in the tube settlers were only marginal at these HRTs. However, the total COD removal efficiency at the two locations differed significantly which indicated that the tube settlers were very effective in removing the suspended fraction of COD (Figure 6.6). Moreover, the fluctuations in efficiencies at various HRTs after the settler were less compared to that before the settler. It can be seen that the settler with $45^{o}$ inclination was more effective in dampening the fluctuations than the settler with $60^{o}$ inclination.

The total $BOD_5$ of the effluent from $R_1$ and $R_2$ was in the range of 17-27 and 18-26 mg/L respectively upto 2.4 h HRT. At 2 h HRT, it was 32

Table 6.1. Performance of $R_1$ Fed with Sucrose-Based Synthetic Wastewater at Various HRTs

| Parameter | | | HRT, h | | | | |
|---|---|---|---|---|---|---|---|
| Name | | Unit/Data type | 5 | 4 | 3 | 2.4 | 2 |
| Influent COD | | mg/L Average ± σ | 485±7 | 480±8 | 488±6 | 485±6 | 486±5 |
| Space loading rate g | | COD/L.d | 2.33 | 2.88 | 3.90 | 4.85 | 5.83 |
| Reactor sludge VSS concentration | | g/L | 11.24 | 11.9 | 13.35 | 11.79 | 10.38 |
| VSS/TSS ratio | | | 0.31 | 0.38 | 0.45 | 0.55 | 0.59 |
| Sludge loading rate | | g COD/ g VSS.d | 0.21 | 0.24 | 0.29 | 0.41 | 0.56 |
| Effluent COD (Efficiency) | Port No. 1 Soluble | mg/L Average ± σ ( % ) | 23±1 (95) | 22±2 (95) | 26±3 (95) | 24±2 (95) | 42±3 (91) |
| | Port No. 2 Soluble | | 25±2 (95) | 27±6 (94) | 29±5 (94) | 30±3 (94) | 67±4 (86) |
| | Port No. 1 Total | | 55±4 (89) | 46±5 (90) | 60±5 (88) | 58±4 (88) | 78±4 (84) |
| | Port No. 2 Total | | 92±15 (81) | 78±15 (84) | 144±12 (70) | 103±12 (79) | 165±12 (66) |
| Closeness to ideal system in total COD removal | | % | 94 | 95 | 93 | 93 | 92 |
| Infl. $BOD_5$ | | mg/L | 467 | 462 | 470 | 465 | 465 |
| Effl. $BOD_5$ | Soluble | | 11 | 8 | 8 | 8 | 16 |
| | Total | | 19 | 17 | 26 | 27 | 32 |

contd...

Table 6.1 (continued)

| Parameter | | HRT, h | | | | |
|---|---|---|---|---|---|---|
| Name | Unit/Data type | 5 | 4 | 3 | 2.4 | 2 |
| Space substrate removal rate | g COD/L.d | 2.21 | 2.74 | 3.71 | 4.61 | 5.30 |
| Specific substrate removal rate | g COD/ g VSS.d | 0.2 | 0.23 | 0.28 | 0.39 | 0.51 |
| Methane recovery rate-NTP | mL/L.d Average ± $\sigma$ | 431±13 | 612±8 | 732±21 | 900±63 | 1076±56 |
| Specific methane production rate | g $CH_4$-COD/ g VSS.d | 0.13 | 0.18 | 0.20 | 0.27 | 0.37 |
| Effluent TSS | mg/L Average ± $\sigma$ | 51±5 | 45±7 | 80±6 | 67±5 | 60±5 |
| Effluent VSS | mg/L Average ± $\sigma$ | 41±4 | 29±4 | 28±5 | 30±3 | 32±6 |
| Wasted sludge VSS | mg/L eff. | 49 | 36 | 30 | 38 | 44 |
| VSS/TSS ratio | | 0.37 | 0.46 | 0.38 | 0.58 | 0.58 |
| BSRT | d | 26 | 30 | 29 | 17 | 12 |
| Specific methanogenic activity | g $CH_4$-COD/ g VSS.d | 0.86 | 0.90 | 0.93 | 0.97 | 1.02 |
| Temperature | $^oC$ | 34 | 35 | 30 | 30 | 29 |

Table 6.2 (continued)

| Parameter | | HRT, h | | | | |
|---|---|---|---|---|---|---|
| Name | Unit/Data type | 5 | 4 | 3 | 2.4 | 2 |
| Space substrate removal rate | g COD/L.d | 2.19 | 2.74 | 3.67 | 4.61 | 5.31 |
| Specific substrate removal rate | g COD/ g VSS.d | 0.21 | 0.23 | 0.27 | 0.41 | 0.52 |
| Methane recovery rate-NTP | mL/L.d Average ± $\sigma$ | 413±12 | 585±27 | 745±42 | 869±51 | 1042±70 |
| Specific methane production rate | g $CH_4$-COD/ g VSS.d | 0.14 | 0.17 | 0.20 | 0.28 | 0.36 |
| Effluent TSS | mg/L Average ± $\sigma$ | 68±7 | 49±5 | 75±8 | 60±6 | 80±8 |
| Effluent VSS | | 52±5 | 28±4 | 28±5 | 26±4 | 34±5 |
| Wasted sludge VSS | mg/L eff | 42 | 38 | 32 | 44 | 37 |
| VSS/TSS ratio | | 0.36 | 0.39 | 0.47 | 0.54 | 0.57 |
| BSRT | d | 24 | 30 | 28 | 16 | 12 |
| Specific methanogenic activity | g $CH_4$-COD/ g VSS.d | 0.90 | 1.00 | 1.03 | 1.09 | 1.08 |
| Temperature | $^{o}C$ | 34 | 35 | 30 | 30 | 29 |

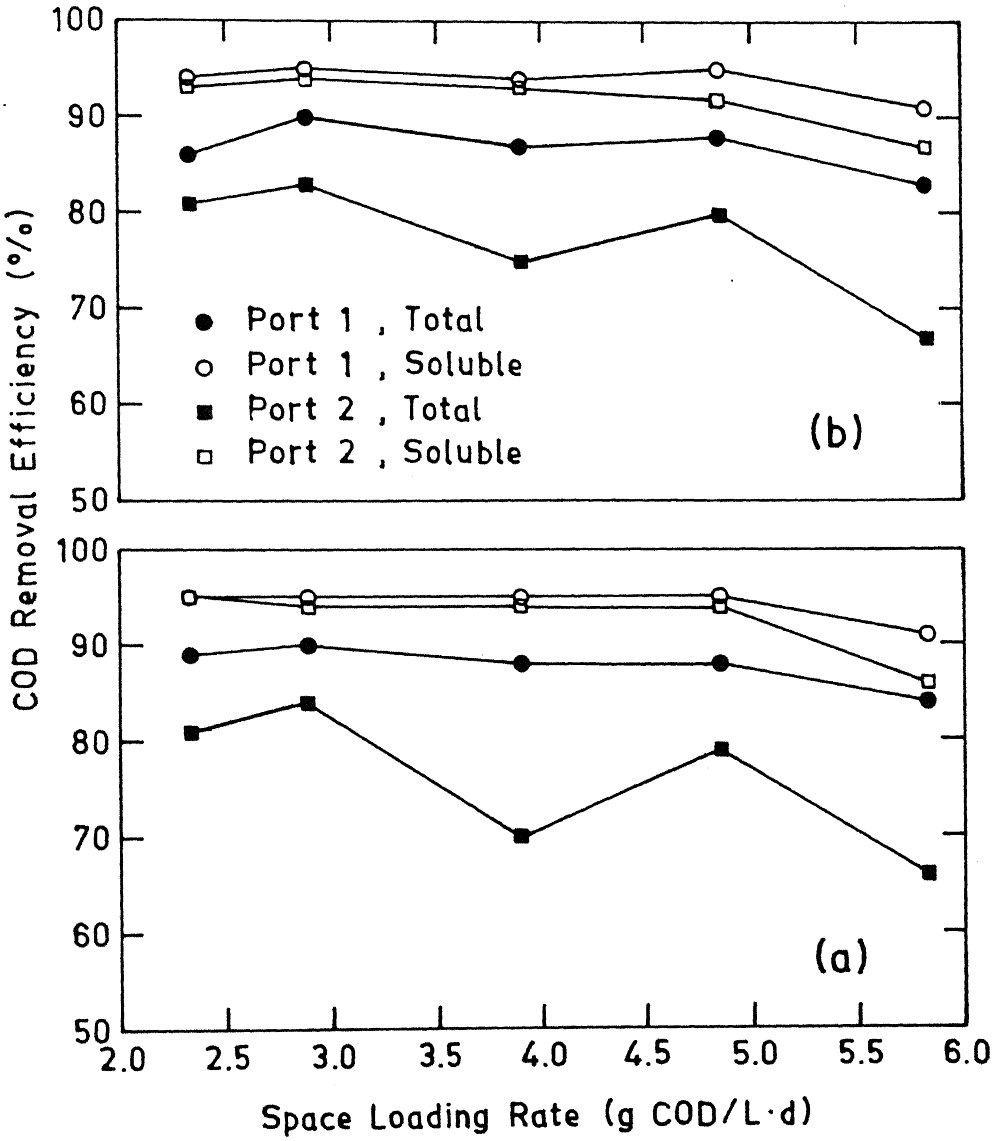



Fig. 6.6. Steady State Reactor Performance in Terms of Soluble COD Removal for Sucrose-Based Wastewater (a) $R_1$ (b) $R_2$.

and 35 mg/L respectively. The soluble $BOD_5$ was in the range of 6-17 mg/L over the entire range of loading.

### 6.3.2 Reactor VSS and Effluent VSS concentration

Figure 6.7a shows the variation of reactor VSS and effluent VSS concentration against the loading rates. The reactor VSS increased gradually with the space loading, reaching a maximum of 13.35 and 13.38 g/L in $R_1$ and $R_2$ respectively at a loading rate of 3.9 g COD/L.d corresponding to the HRT of 3 h. Thereafter, the VSS content in both the reactors reduced gradually. The increase in sludge content at HRT 4 and 3 h compared to that at 5 h indicates that the sludge was getting denser during this period as the sludge occupied same volume of the reactor at all HRTs. This could be the effect of granulation which was observed at HRT 4, as the microbes are more densely packed in granules than in flocculant sludge. Moreover, on maturation the granules become more compact (Hulshoff Pol 1989). However, at HRTs less than 3 h, due to the higher hydraulic loading and gas production rates, the bed expansion was more resulting in a higher rate of withdrawal of sludge than accumulation rate.

The maximum VSS concentration in the effluent was observed at an HRT of 5 h (2.33 g COD/L.d) in both the reactors (Figure 6.7a). At lower HRTs, the VSS concentration remained more or less constant, with a slight fluctuation in $R_2$ (Table 6.1 and 6.2). The effluent TSS concentration in $R_1$ and $R_2$ were in the range of 45-80 and 49-80 mg/L over the range of HRTs studied. The highest VSS/TSS ratio of the effluent from both the reactors (>0.76) was also observed at 5 h HRT.

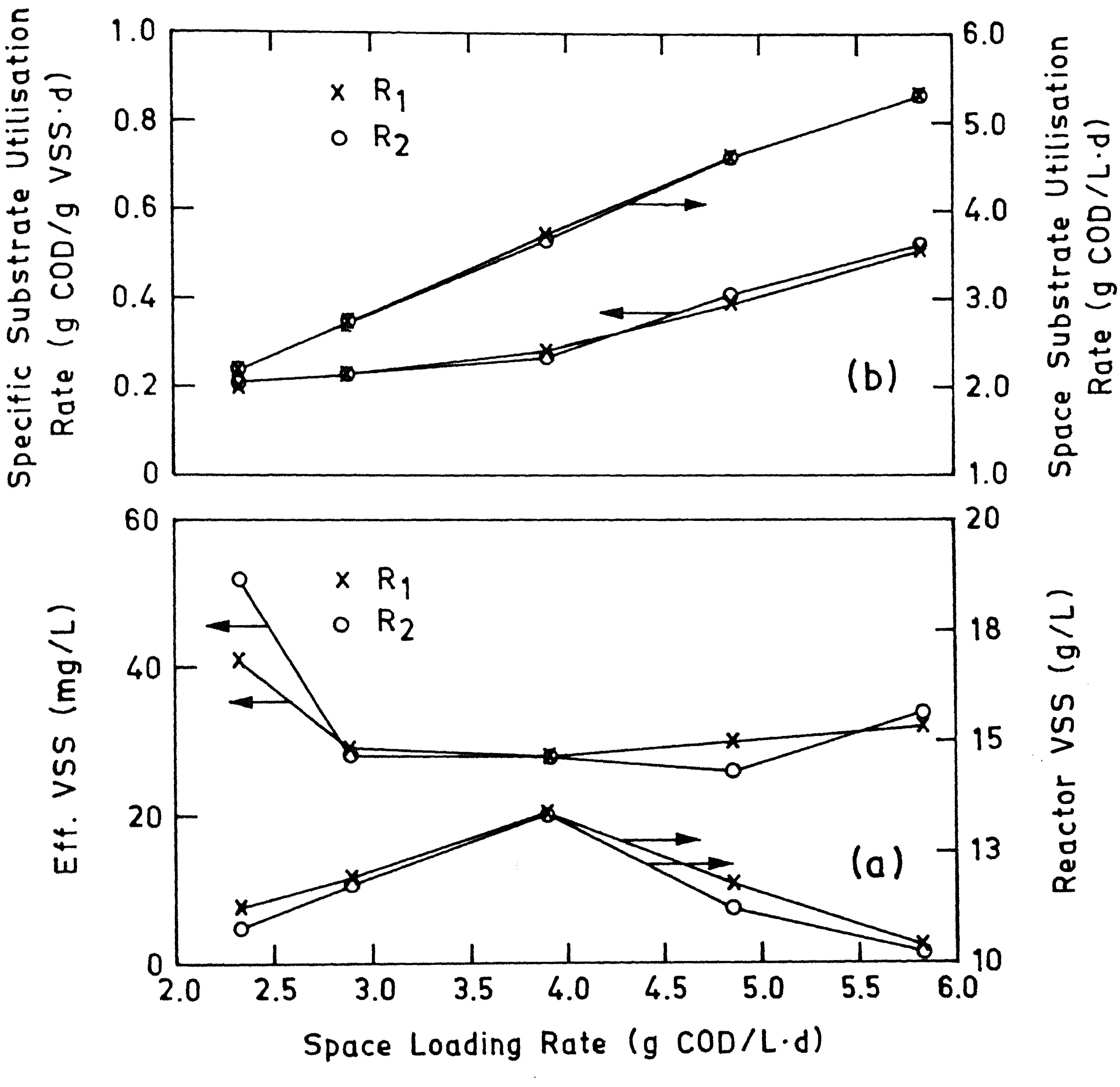



Fig. 6.7. Steady State Reactor Performance in Terms of VSS and Substrate Utilization Rate for Sucrose-Based Wastewater .

Possibly, the discrete microbes which could not be removed in the settler unit might have been more at this HRT. With the development of granular sludge at 4 h HRT, the release of very fine biomass from the sludge bed might have reduced. Another reason may be the capturing of any discrete organism, released even after granulation, by the attached wall growth on the tube settler due to prolonged operation. This microbial growth on the tube was evident by slightly higher contribution in soluble COD removal at lower HRTs by the settler (Figure 6.6).

### 6.3.3 Substrate Utilisation Rate

The variation of specific substrate utilization rate (g COD/g VSS.d) and space substrate utilization rate (g COD/L.d) with space loading rate is shown in Figure 6.7(b). The increase in specific substrate utilization rate indicated the over all improvement in the microbial activity as the loading rate increased. It can be noted that the increase in specific substrate utilisation rate beyond a loading rate of 3.9 g COD/L.d, was distinctly at a different pace. This higher pace of microbial activity at higher food to microorganism (F/M) ratio, due to both the increase in loading and decrease in biomass concentration (Figure 6.7a), indicates the approaching of microbial growth rate closer to the maximum rate.

The space substrate utilization rate increased from about 2.2 to 5.3 g COD/L.d as the loading rate increased from 2.33 to 5.83 g COD/L.d corresponding to the decrease in HRT from 5 h to 2 h. A lower rate of increase in substrate utilisation rate was observed beyond 2.4 h HRT (Fig. 6.7b). While 96% of the increased load was destroyed till 2.4 h

HRT, it was only 71% at 2 h. This indicated that the effect due to reduction in biomass between 2.4 and 2 h HRT has out-competed the increase in specific substrate utilization rate. This resulted in lesser soluble COD removal efficiency at 2 h than at 2.4 h (Fig. 6.6).

### 6.3.4 The Reactor VSS/TSS Ratio and BSRT

Figure 6.8(a) shows the variation of reactor sludge VSS/TSS ratio with the loading rate. At the lowest loading rate of 2.33 g COD/L.d (5 h HRT), the VSS/TSS ratio in $R_1$ and $R_2$ were 0.31 and 0.34 respectively. As the loading rate increased, the VSS fraction in the sludge increased and the ratios were 0.59 and 0.58 at HRT 2 h in $R_1$ and $R_2$.

This increase in VSS/TSS ratio with decrease in HRT might have resulted from the continuous enrichment of the reactor sludge, originally at a lower VSS/TSS ratio, with the newly synthesized biomass during the prolonged reactor operation at various loading rates. The increase in microbial growth rate due to the increase in F/M ratio and the daily sludge wasting from port No.2 effecting the removal of TSS from the reactor are factors that accelerate the microbial enrichment.

Figure 6.8(a) also illustrates the decrease in Biological Sludge Retention Time (BSRT) with increase in loading rate (decrease in HRT). The BSRT was calculated based on the sludge lost with effluent and the sludge wasted from port No. 2. The sludge wasted is expressed in mg of VSS per litre of waste treated (Table 6.1 and 6.2). It can be seen that in both the reactors, the sludge wasted was significant compared to the sludge lost in the effluent. Moreover, there was hardly any increase in

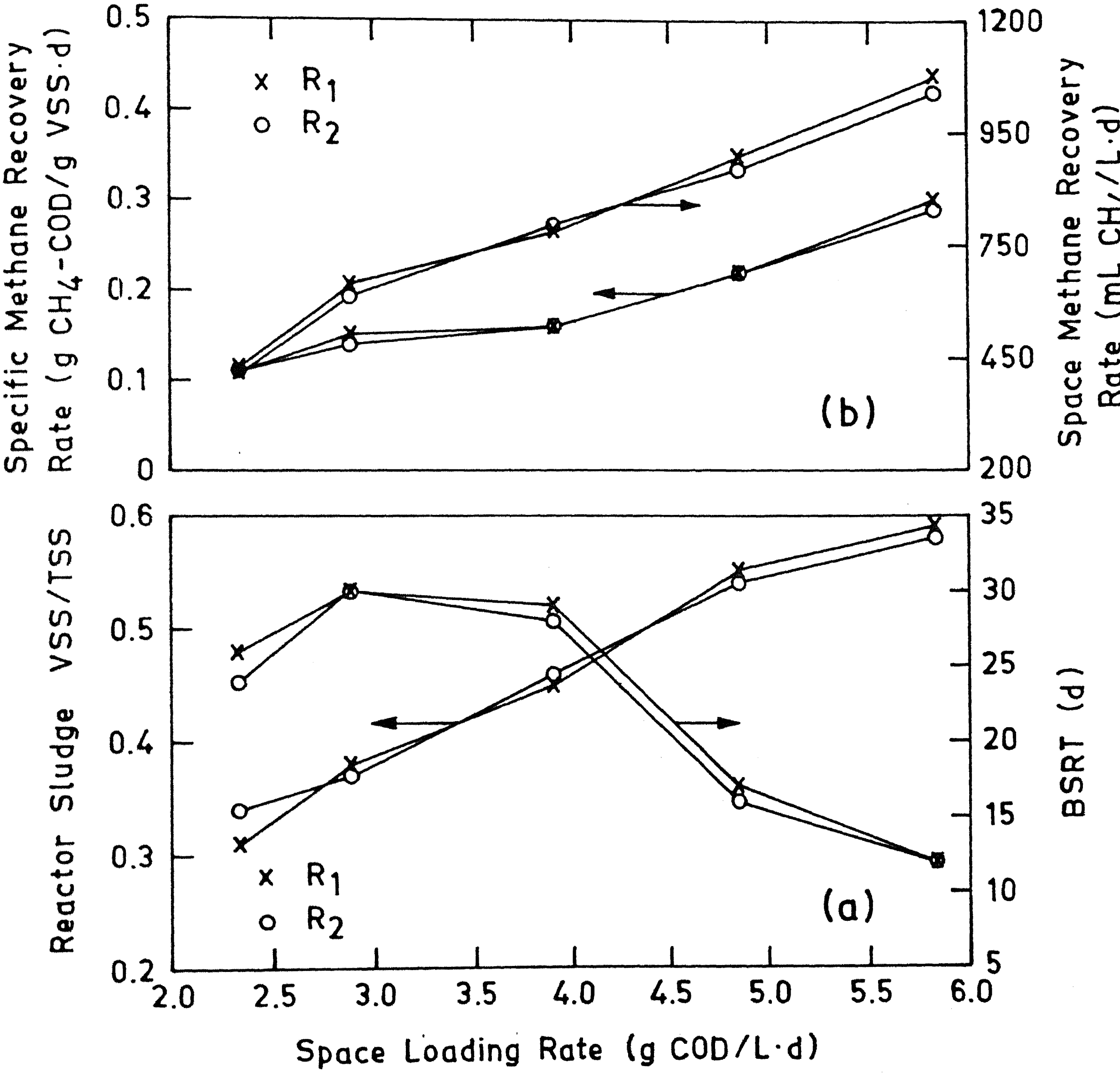



Fig. 6.8. Steady State Reactor Performance in Terms of VSS/TSS, BSRT and Methane Recovery Rate for Sucrose-Based Wastewater.

effluent VSS concentration with decrease in HRT. Therefore the decrease in BSRT with HRT can mainly be attributed to increase in flow rate and the sludge wasting from port No. 2.

### 6.3.5 Methane Production

The methane content of the biogas was found to be 73-78% over the range of HRT studied. The increase in methane recovery rate per unit reactor volume (space methane recovery rate) and specific methane recovery rate, without accounting the methane loss in the effluent, with the COD loading rate is shown in Figure 6.8b. The variation in methane recovery rates, both space and specific, followed a trend almost similar to that of the substrate utilisation rates (Figure 6.7b) except between 5 h and 4 h HRT. An observed higher pace of increase at these HRTs indicated that relatively a higher fraction of the substrate utilized was going for energy production than cell synthesis. It may be recalled that there was an increase in BSRT at this stage.

The specific methanogenic activity (SMA) of the sludge from both the reactors $R_1$ and $R_2$ varied within a narrow range of 0.86-1.02 and 0.9-1.09 g $CH_4$-COD/g VSS.d respectively over the entire range of loading rates employed. However, there was a significant variation in SMA along the height of the sludge bed. In general, the activity was the highest at the bottom and it reduced towards the top of the bed. This variation was more prominent at lower loading rates. This might have been due to the microbes at bottom of the reactors confronting a higher substrate flux for a longer duration at higher HRTs.

The methane lost with the effluent in dissolved form is considerable especially for low strength wastewaters (Kobayashi et al., 1983; Lettinga, 1992). For a reactor temperature of 30°C and 75% of methane content in the gas phase, the saturation methane concentration at STP is approximately 21 mL/L in water. In the present investigation, at an HRT of 3 h, approximately 1540 mL of methane at STP would have been lost in the effluent daily whereas the recovered methane was 6825 mL/d. As this loss is considerable, a correction equivalent to the saturation concentration, was applied to the recovery rate at each HRT to get the specific methane production rate (Table 6.1 and 6.2).

Figure 6.9 illustrates the variation of specific methane production rate (SMPR) with the specific substrate utilization rate. The slope of the best fit linear line for the data from both the reactors was 0.70. This slope indicated that of all the COD removed, 70% was converted to methane and the rest was presumably, converted to biomass. Based on the steady-state VSS concentration in the effluent and VSS wasted every day, the VSS yield per g COD removed was estimated for $R_1$ and $R_2$ for each HRTs. The average of this yield coefficient was 0.16 g VSS/g COD removed. Assuming a VSS-COD/VSS ratio of 1.4, the sludge yield coefficient amounted to 0.22 g VSS-COD/g COD removed which is close to the maximum cell yield coefficient of 0.28 reported by Young and McCarty (1969) for carbohydrate substrate. During granulation experiments with sucrose rich wastewater Hulshoff Pol (1989) has observed a sludge yield of 0.13 g VSS-COD/g.COD removed. This value was calculated based on the

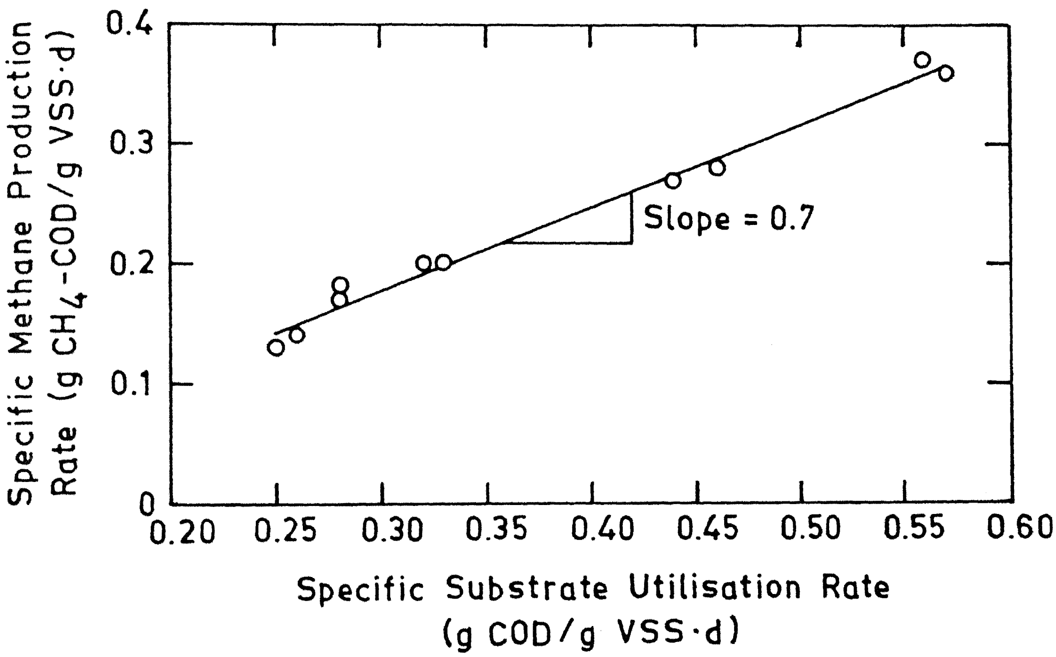



Fig. 6.9. Specific Methane Production Rate at Various Specific Substrate Utilisation Rates for Sucrose-Based Wastewater.

sludge accumulation in the reactor without considering the biomass lost with effluent which could be quite considerable.

Methane production, in general, surpasses 90% of the COD processed due to low yield coefficient (Henze and Harremoes, 1985; Henze et al., 1995). Compared to this, it appears that less fraction of the COD destroyed was converted to methane in the present investigation. The yield coefficient is dependent on F/M ratio. At higher F/M ratio more of the substrate goes for cell synthesis yielding less methane. Fang and Chui (1993), and Yang and Anderson (1993) have observed lower fraction of substrate converted to methane at higher F/M ratio. On the other hand, at low F/M ratio, higher fraction of COD goes for production of energy. Based on studies conducted with low strength (300 mg/L COD) sucrose synthetic wastewater using hybrid reactors, Droste et al. (1987) have reported a maximum conversion of COD to methane with no net growth below a loading rate of 1.3 g COD/L.d. The corresponding F/M ratio was about 0.05 g COD/g VSS.d. At a loading rate of 0.09 g COD/g VSS.d, above 80% of COD used was converted to methane. It could be also noted that at these stages of higher energy yield, the reactors were operating at a BSRT of 80 d or above.

In the present investigation, the specific substrate loading rate (F/M ratio) was in the range of 0.21-0.57 g COD/g VSS.d. The BSRT of the reactors ranged from 30-12 d during the study. As the BSRT reduces, the microbial system enters to the exponential growth phase where the growth will be close to the maximum rate. At this stage, the fraction

of substrate going for cell synthesis will be more and methane yield will be less.

The primary aim of regular sludge wasting from port No. 2 was to prevent the sludge from occupying the settler zone and thus keep the functional aspect of settler zone constant throughout the study. However, this regular wasting of sludge has also contributed to the decrease in BSRT and to the increase in F/M ratio.

The results of the study demonstrated that the reactors could effectively be used to produce effluents of good quality while treating low strength sucrose-based synthetic wastewater. The tube settlers were found to be effective in reducing the suspended fraction of effluent COD. Soluble COD removal in the settler was marginal. The total COD of the effluent was in the range of 46-82 mg/L. The total $BOD_5$ of the effluent from both the reactors was below 30 mg/L even at an HRT of 2.4 h while the influent $BOD_5$ was in the range of 462-470 mg/L.

### 6.4 Secondary Start-up with CERELAC-Based Synthetic Wastewater

The next phase of the study was designed to evaluate the suitability of the UASB reactor with the modified configuration for treatment of wastewater containing complex organic substrates. For this purpose, a synthetic wastewater prepared by using a commercial grade babyfood (CERELAC, NESTLE, INDIA) was used. The composition of this wastewater was considerably different from sucrose wastewater as it contained insoluble carbohydrates, proteins and fats. A "secondary start-up" operation was thus carried out for a period of about one and a half months before attaining a steady-state. During this period of

adaptation, an ecological shift was expected to occur in the granular sludge developed on sucrose wastewater. The schedule of operation adopted and the data on important process parameters of $R_1$ are presented in Fig. 6.10. The performance of reactor $R_2$ during this period was almost similar to that of $R_1$ and hence is not presented.

After completion of the steady-state operation at 2 h HRT with synthetic sucrose wastewater, the HRT was increased to 4 h. The secondary start-up was carried out by gradually replacing sucrose with CERELAC, keeping the total COD of the synthetic wastewater constant. From day 23, CERELAC was the only carbon source in the wastewater. The wastewater was supplemented with other nutrients as outlined in Table 5.1 and required amount of bicarbonate to maintain the reactor pH in the range of 7 to 7.3. The reactor operation was continued at 4 h HRT till day 53 The average total and soluble COD of the wastewater used during 53 days of operation were 476 mg/L and 205 mg/L respectively. The corresponding $BOD_5$ values were 322 mg/L and 163 mg/L. The reactor temperature during this period reduced significantly from 24 to 12.5$^{o}$C.

Figure 6.10(a) shows the step wise increase in percentage of CERELAC in the influent COD during the secondary start up. By day 7, 40% of the influent COD was constituted by CERELAC, and the remaining by sucrose. This combination was maintained till day 11. During this period, the total and soluble effluent COD was about 110 mg/L and 40 mg/L (Fig. 6.10b) which corresponded to a COD removal efficiency of about 77% and 80% respectively. The total gas production rate at this stage was about 4000 mL/d (Fig. 6.10a). To be noted is the enormous

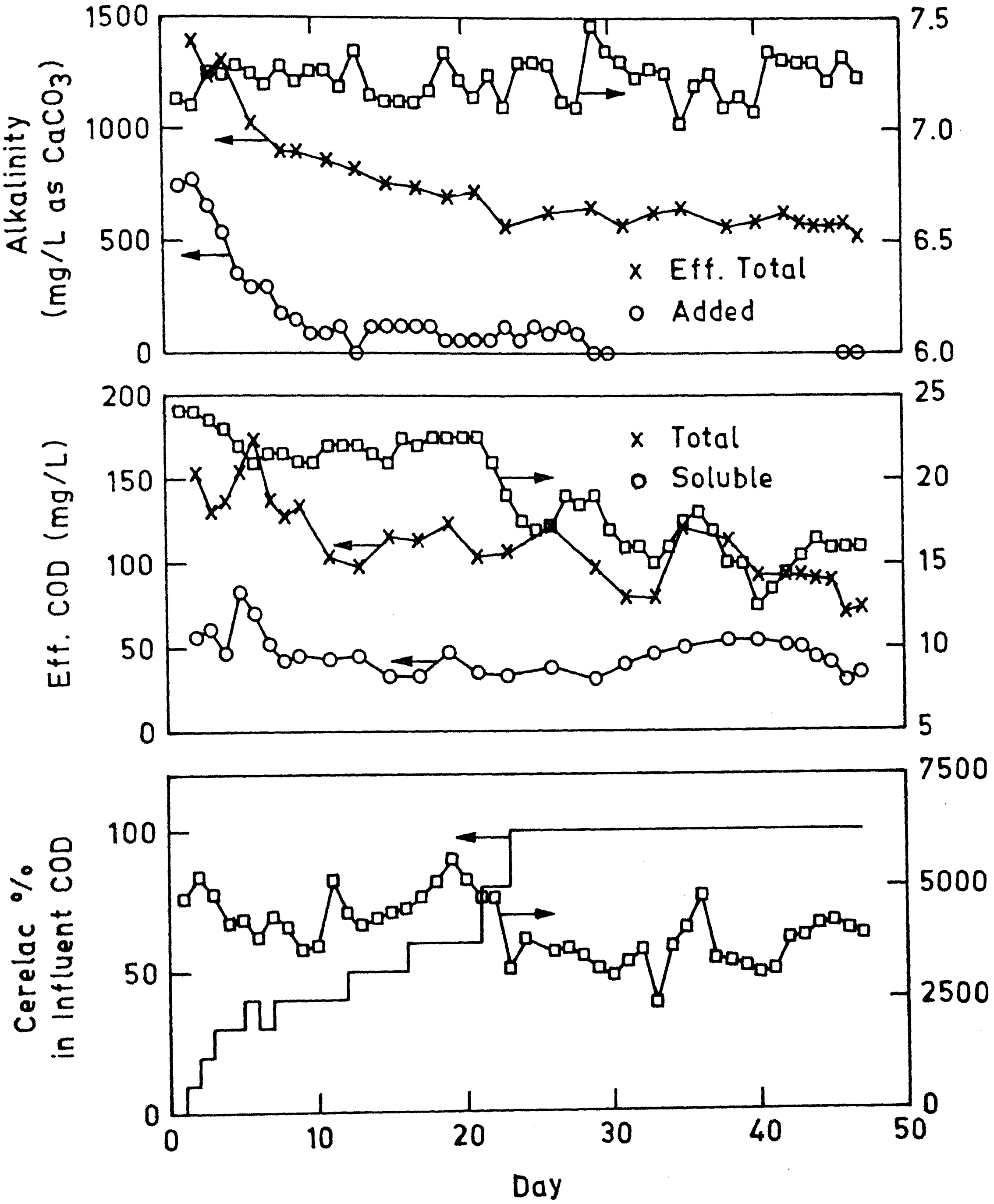



Fig. 6.10. Performance of $R_1$ Fed with Cerelac-Based Wastewater During Secondary Start-up.

reduction in the alkalinity requirement to maintain the reactor pH above 7.1 from about 774 to 120 mg/L as $CaCO_3$ (Fig. 6.10c). The CERELAC percentage in the feed was further increased gradually achieving 80% by day 21. A slight reduction in the effluent soluble COD concentration and a gradual increase in gas production rate was noted from day 11 to 21. The reactor temperature varied within a narrow range of 21-22.5$^{o}$C during this period.

The entire source of carbon in the wastewater was CERELAC from day 23 till the end of this study. A general downward trend in gas production rate could be seen from day 22 to 33 which appeared to be mainly due to fall in reactor temperature from 22.5$^{o}$C to 15$^{o}$C. Concomitantly the gas production reduced from 4735 mL/d on day 22 to 2427 mL/d on day 33. However, from day 37, the gas production rate appeared to be less fluctuating. The effluent COD also was influenced by the fall in temperature, but to a lesser extent. This may be attributed to the removal of suspended fraction of influent COD by physical action which is not significantly affected by the fall in temperature unlike biological stabilisation.

The quantity of bicarbonate required to be added with influent to maintain the reactor pH above 7.1 from day 10 was very nominal, about 120 to 60 mg/L as $CaCO_3$. Bicarbonate addition was completely terminated on day 29. Still, the effluent alkalinity remained more or less constant (520-640 mg/L as $CaCO_3$) till the end. Similar behavior was exhibited by $R_2$.

Ammonia is formed in the reactor during the degradation of proteins present in the waste. This ammonia exists either as ammonium ion ($NH_4^+$) or as dissolved ammonia gas ($NH_3$) in solution. The $NH_4^+$ - $NH_3$ can act as an effective buffering system in anaerobic processes. It appears that the buffering system constituted by the bicarbonate formed during methane generation and the ammonia formed during protein degradation, was sufficient enough to maintain the required pH and alkalinity in both the reactors.

The average of the effluent total COD for three days from day 45 in $R_1$ and $R_2$ was 77 and 85 mg/L which corresponded to the treatment efficiency of 84% and 82% respectively. The soluble COD removal efficiency in both the reactors during this period was above 80%. The self maintenance of reactor pH and alkalinity, combined with the high treatment efficiency at reactor temperature as low as 15°C indicated a good adaptation of the granular sludge to the new wastewater.

### 6.5 Reactor Performance During Steady-state Operation with CERELAC-Based Synthetic Wastewater

On completion of the secondary start-up with synthetic CERELAC wastewater, both the reactors were operated at various HRTs to get PSS data. As the influent COD was kept constant, decrease in HRT resulted in proportionate increase in space loading rate. During the steady-state operation at each HRT, the data on reactor performance was collected for 5 consecutive days. The mode of study was similar to that adopted during synthetic sucrose wastewater feeding. The details of

steady-state performance at 4 and 2 h HRT for $R_1$ and 4, 2 and 1 h HRT for $R_2$ are summarized in Table 6.3.

### 6.5.1 COD and BOD Removal Efficiencies

In order to get the treatment efficiency that can be achieved by an ideal settling system, the COD removal efficiency based on influent total and filtered effluent, hereafter referred as COD(TI-FE), was also calculated apart from total and soluble COD removal efficiency. On comparison of the removal efficiency based on total COD with COD(TI-FE) (Table 6.3) it can be seen that both the reactors were about 90% close to the ideal system at 4 h and 2 h HRT.

Figure 6.11(a) shows the total COD removal efficiency of both reactors at various space loading rates. The efficiency in $R_1$ and $R_2$ based on effluent samples from port No. 1, at loading rates 5.64 and 2.86 g COD/L.d (HRTs 2 and 4 h) were in the range of 81-83%, whereas the efficiencies based on settled samples from port No. 2 were in the range of 59-63%. However, only a marginal improvement in soluble COD removal efficiency of 75% (port No. 2) to 80% (port No. 1) due to the tube settlers was observed indicating minimal biological activity in this zone. Hence, the significant increase in total COD removal efficiency, from port No. 2 to 1 shows that the tube settlers were very effective in retaining suspended solids in the reactor.

The total $BOD_5$ of the effluent from $R_1$ and $R_2$ were 28 and 32 mg/L respectively at 4 h HRT. The corresponding values at 2 h HRT were 32 and 35 mg/L. The soluble $BOD_5$ at these two HRTs were in the range of

Table 6.3. Performance of $R_1$ and $R_2$ Fed with CERELAC-Based Synthetic Wastewater at Various HRTs

| Parameter | | | HRT, h | | | | |
|---|---|---|---|---|---|---|---|
| Name | | Unit/Data type | $R_1$ 4 | $R_1$ 2 | $R_2$ 4 | $R_2$ 2 | $R_2$ 1 |
| Influent COD | Total | mg/L | 476±6 | 470±8 | 476±6 | 470±8 | 472±6 |
| | Soluble | Average ± σ | 205±6 | 200±8 | 205±6 | 200±8 | 205±9 |
| Space loading rate | | g COD/L.d | 2.86 | 5.64 | 2.86 | 5.64 | 11.33 |
| Reactor sludge VSS concentration | | g/L | 8.86 | 7.0 | 8.6 | 6.63 | 6.14 |
| VSS/TSS ratio | | | 0.59 | 0.66 | 0.60 | 0.66 | 0.72 |
| Sludge loading rate | | g COD/ g VSS.d | 0.32 | 0.81 | 0.33 | 0.85 | 1.85 |
| Effluent COD (Efficiency) | Port No. 1 Soluble | mg/L Average ± σ ( % ) | 37±3 (82) | 45±4 (78) | 37±3 (82) | 43±4 (79) | 161±8 (21) |
| | Port No. 2 Soluble | | 51±8 (75) | 55±5 (73) | 46±6 (78) | 59±11 (71) | - |
| | Port No. 1 Total | | 83±7 (83) | 88±5 (81) | 86±7 (82) | 88±8 (81) | 252±10 (47) |
| | Port No. 2 Total | | 178±35 (63) | 179±21 (62) | 194±23 (59) | 190±21 (60) | - |
| Efficiency | Port No. 1 COD (TI-FE) | % | 92 | 90 | 92 | 91 | 66 |
| Closeness to ideal system in total COD removal | | % | 90 | 90 | 89 | 89 | 71 |
| Infl. $BOD_5$ | Soluble | mg/L | 163 | 160 | 163 | 160 | - |
| | Total | | 322 | 320 | 322 | 320 | - |
| Effl. $BOD_5$ | Soluble | | 20 | 18 | 15 | 18 | - |
| | Total | | 28 | 32 | 32 | 35 | - |

contd...

Table 6.3 (continued)

| Parameter | | HRT, h | | | | |
|---|---|---|---|---|---|---|
| Name | Unit/Data type | $R_1$ 4 | $R_1$ 2 | $R_2$ 4 | $R_2$ 2 | $R_2$ 1 |
| Space substrate removal rate | g COD/L.d | 2.36 | 4.58 | 2.35 | 4.57 | 5.33 |
| Specific substrate removal rate | g COD/ g VSS.d | 0.27 | 0.65 | 0.27 | 0.69 | 0.87 |
| Methane recovery rate-NTP | mL/L.d Average ± $\sigma$ | 336±5 | 608±22 | 331±15 | 600±8 | 458±58 |
| Specific methane production rate | g $CH_4$-COD/ g VSS.d | 0.17 | 0.36 | 0.17 | 0.38 | 0.44 |
| Effluent TSS | mg/L Average ± $\sigma$ | 35±8 | 47±6 | 43±10 | 41±12 | 201±27 |
| Effluent VSS | mg/L Average ± $\sigma$ | 25±4 | 38±5 | 29±5 | 26±7 | 130±18 |
| Wasted sludge VSS | mg/L eff | 46 | 41 | 43 | 44 | 24 |
| VSS/TSS ratio | | 0.62 | 0.63 | 0.55 | 0.66 | 0.67 |
| BSRT | d | 21 | 7 | 20 | 8 | 1.7 |
| Specific methanogenic activity | g $CH_4$-COD/ g VSS.d | 0.82 | 1.6 | 0.84 | 1.56 | - |
| Temperature | $^{o}C$ | 15 | 23 | 15 | 23 | 26 |

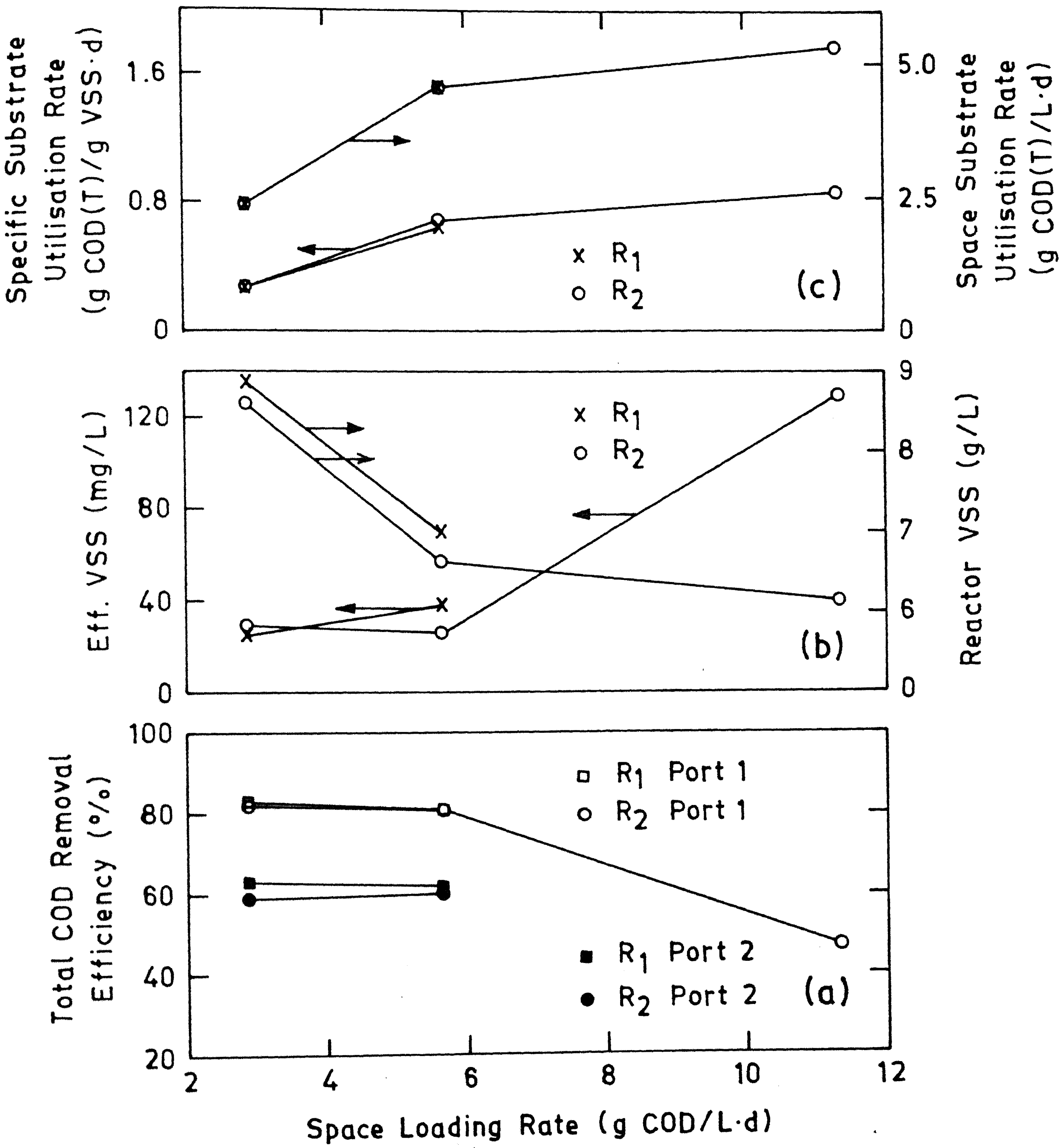



Fig. 6.11. Steady State Reactor Performance in Terms of Total COD Removal, VSS and Substrate Utilisation Rate for Cerelac-Based Wastewater.

15-20 mg/L in the two reactors. It is to be noted that at 4 h and 2h HRT the reactor temperature was $15^{o}C$ and $23^{o}C$ respectively.

After completion of the steady-state performance evaluation at 2 h HRT, operation of reactor $R_2$ was further continued at a space loading rate of 11.33 g COD/L.d (1 h HRT) for 15 days. During this period sludge washout and deterioration of effluent quality were observed. The soluble and total COD removal efficiencies were 21% and 47% respectively. These low COD removal efficiencies indicated a reduction both in waste stabilization rate in the sludge zone as well as suspended solids removal in the settler. Even under these conditions, the reactor was 71% close to an ideal system.

### 6.5.2 Effluent VSS and Reactor VSS Concentration

Figure 6.11(b) shows the variations in effluent and reactor VSS concentrations with respect to space loading rates. When the loading rate was increased from 2.86 to 5.64 g COD/L.d (HRT 4 to 2 h) the effluent VSS concentration remained more or less constant in the range of 25-38 mg/L. TSS concentrations in the effluent in both reactors were less than 50 mg/L. However, higher VSS and TSS concentrations (130 and 201 mg/L) were observed at 1 h HRT in $R_2$ indicating the escape of suspended solids.

The reactor VSS concentrations were 8.6, 6.63 and 6.14 g/L corresponding to the loading rates of 2.86, 5.64 and 11.33 g COD/L.d (HRTs 4, 2 and 1 h) respectively for reactor $R_2$. This decrease in reactor VSS concentration with HRT indicates that the sludge removal rate (sum of VSS wasted and VSS in the effluent) was more than the

sludge production rate. However, the effect of sludge wasting was significantly higher than that of VSS loss in the effluent (Table 6.3). However, the loss of VSS through the effluent had significantly influenced the reactor VSS concentration at 1h HRT.

### 6.5.3 Substrate Utilization Rate

The variation of specific substrate utilization rate (g COD (T)/g VSS.d) and space substrate utilization rate (g COD (T)/L.d) against space loading rate is shown in Fig. 6.11(c). The increase in specific substrate utilization rate with F/M ratio, due to increase in loading rate as well as decrease in reactor VSS concentration, indicated the increase in microbial growth rate. A lower increase in specific substrate utilization rate between 2 h and 1 h HRT compared to that between 4 h and 2 h and the lower reactor VSS concentration at 1 h HRT had resulted in a lower increase in space substrate utilization rate between 2 h and 1 h HRT.

### 6.5.4 The Reactor VSS/TSS Ratio and BSRT

Figure 6.12(a) illustrates the variation of the reactor sludge VSS/TSS ratio and BSRT with space loading rate. As the HRT was reduced from 4h to 2h, the VSS/TSS ratio in both the rectors increased from about 60 to 66%. In reactor $R_2$ the VSS/TSS ratio was 72% at 1 h HRT.

The continuous enrichment of the reactor sludge, with the newly synthesized biomass during the prolonged reactor operation at various loading rates, the daily sludge wasting from port No.2 effecting the removal of TSS from the reactor and accumulation of the physically

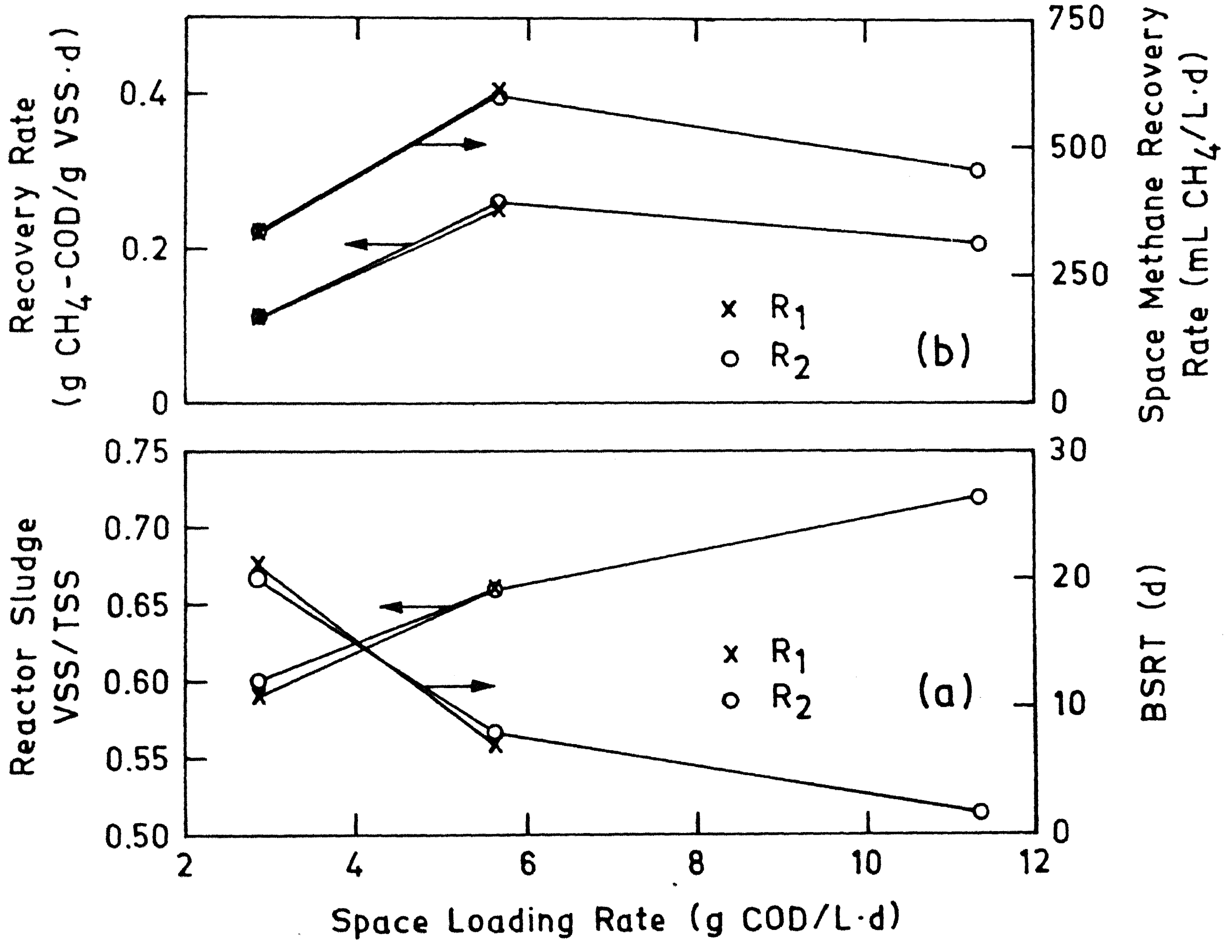



Fig. 6.12. Steady State Reactor Performance in Terms of VSS/TSS, BSRT and Methane Recovery Rate for Cerelac-Based Wastewater.

trapped suspended solids of the influent are factors that contribute to the observed increase in VSS/TSS ratio along with the decrease in HRT. The increase in microbial growth rate due to the increase in F/M ratio, that too with a nutritionally more balanced substrate might have contributed to the microbial enrichment.

The reduction in BSRT with increase in loading rate is shown in Fig. 6.12(a). In reactor $R_2$, the BSRT reduced from 20 to 8 d as the HRT reduced from 4 h to 2 h and at 1 h it further reduced to 1.7 d.

The BSRT was calculated based on the sludge lost with effluent and the sludge wasted from port No. 2. The sludge wasted is expressed in mg of VSS per litre of waste treated (Table 6.3). It can be seen that in both the reactors, at 4 h and 2 h HRT, the sludge wasted was significant compared to the sludge lost in the effluent. So the low BSRTs at these two HRTs was mainly due to the sludge wastage from port No. 2. This sludge wasting was necessary to keep the effective settler length constant. However, the very low BSRT at 1 h HRT may be attributed to the high VSS loss with the effluent, obviously due to the limitation of the settler in retaining the biomass at this loading rate.

### 6.5.5 Methane Production

The methane content in the biogas was in the range of 70-77% for the loading rates studied. The methane percentage reduced with the increase in loading rates in both the reactors. The variation of specific methane recovery rate and space methane recovery rate with loading rate is given in Fig. 6.12(b). The methane recovery rates were higher at the loading rate of 5.64 g COD/L.d (HRT 2 h) compared to that

at 2.86 g COD/L.d (HRT 4 h) in both the reactors, possibly due to the higher substrate utilization rate at 2 h HRT. However, at the loading rate of 11.33 g COD/L.d (HRT 1 h) in $R_2$ the recovery rate decreased. This might have resulted from the higher loss of methane per day through the effluent at higher hydraulic loading rates as the specific methane production rate, which accounts also the methane lost with the effluent, was high at 1 h HRT compared to that at 2 h (Table 6.3).

The average SMA of the sludge from the two reactors at 4 h and 2 h HRTs was 0.83 and 1.6 g $CH_4$-COD/g VSS.d. The significantly high SMA at 2 h HRT compared with the activity of 2.4 g $CH_4$-COD/g VSS.d observed with acetate enrichment culture (Valcke and Verstraete, 1983), indicated a high percentage of acetoclastic methanogens in the sludge granules. Prolonged acclamation of sludge in the CERELAC-based wastewater containing a wide variety of nutrients, might have promoted a better growth and accumulation of the fastidious acetoclastic methanogens. Valcke and Verstraete (1983) reports that methanogens growing logarithmically can exhibit specific activities surpassing those of stationary phase cells. So the relatively low BSRT in the reactor (around 7.5 d ) resulting in logarithmic growth of the acetogens which have a generation time of 2-10 d, also might have contributed to the high activity of the sludge.

The specific methane production rate (SMPR) which accounts for $CH_4$ in the biogas and dissolved in the effluent as a function of specific substrate utilization rate (based on effluent total COD) is shown in Fig. 6.13. The slope of the best fit linear line for data of both the

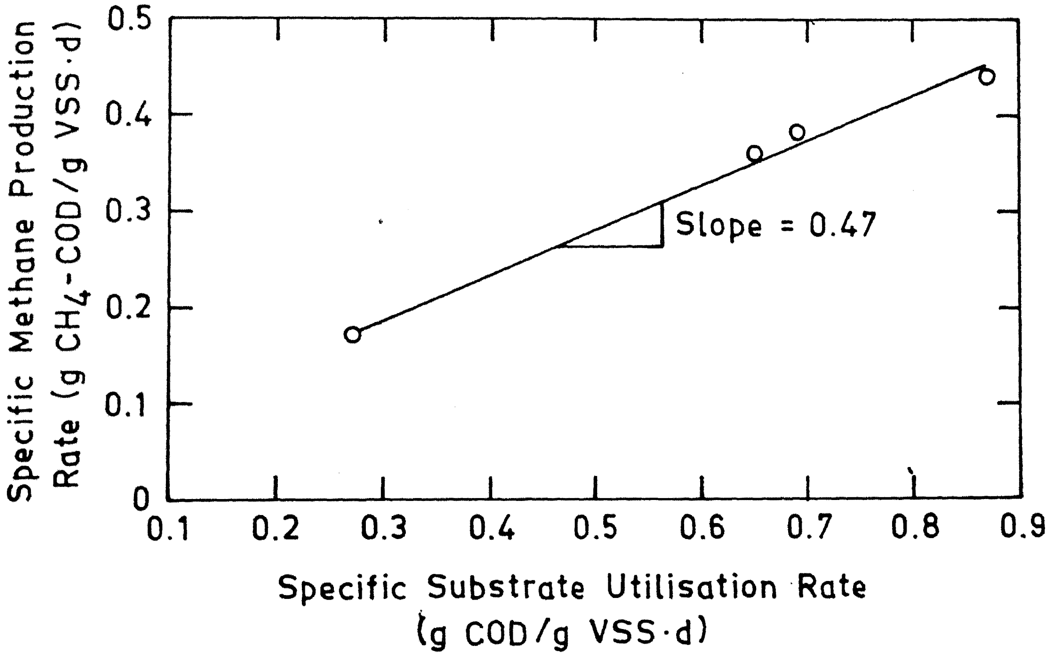



Fig. 6.13. Specific Methane Production Rate at Various Specific Substrate Utilisation Rates for Cerelac-Based Wastewater.

reactors was 0.47. This indicated that only 47% of the COD removed was accounted by methane. The low methane yield may partially be attributed to the low BSRTs and the related high F/M ratios that were prevailing in the reactor. A comparison of the ratio of SMPR and specific substrate utilisation rate (Table 6.3) for various HRTs shows that lower the BSRT lesser was the methane yield. At these conditions a smaller fraction of substrate will go for methane production. Scum formation at the liquid gas interface due to the presence of fat and protein in CERELAC may constitute some fraction of the COD removed.

The possibility of methane lost through the effluent, over and above the saturation concentration cannot be ruled out. Pauss et al. (1990) have reported chances of over-concentration of dissolved methane up to 12 times the saturation concentration in the liquid phase of anaerobic reactors due to mass transfer limitations. A fraction of COD removed may also be accounted for the energy lost as heat and work to the aqueous system.

Considering the steady-state VSS wastage and that lost in the effluent the VSS yield per g of COD removed based on effluent COD(TI-FE) was estimated for $R_1$ and $R_2$ at each HRTs. Assuming a VSS-COD/VSS ratio of 1.4 (Hulshoff Pol, 1989) the average sludge yield coefficient amounted to 0.23 g VSS-COD/g COD removed. The lower methane yield and relatively higher sludge yield indicated that a fraction COD removed has to be accounted for the precipitated or adsorbed solids or fat. The effect of low reactor temperature where endogenous respiration is low can also result in a high sludge yield.

Results of this study demonstrated that the modified UASB reactor configuration could effectively be employed to treat low-strength synthetic wastewater containing complex organic substrate at ambient temperature conditions. The granular sludge developed on sucrose wastewater could be easily adapted to the CERELAC-based wastewater. The total COD of the effluent at 4 h and 2 h HRT was in the range of 83-88 mg/L. The treatment efficiency was about 90% close to a system with an ideal sedimentation system at these HRTs. The effluent total $BOD_5$ was in the range of 28-35 mg/L. At 1 h HRT the settler consisting of tubes of 2 cm diameter was found to be less effective in gas-liquid-solid separation.

**6.6 Treatment of Raw Domestic Wastewater**

The final phase of the study was meant to assess the suitability of the reactor configuration to treat raw domestic wastewater at low HRTs. The wastewater collected from a sump well in the residential area of Indian Institute of Technology Kanpur (India) was used as the feed and its main characteristics are given in Table 5.4.

As the reactors $R_1$ and $R_2$ with settler inclination of $45^o$ and $60^o$ respectively exhibited only marginal difference in the performance while treating synthetic sucrose and CERELAC wastewaters, no effort was made to compare the performance of these two reactors at same HRTs. Reactor $R_1$ was operated at 3 h and 1 h HRT and $R_2$ was operated at 2 h HRT simultaneously. After collection of 5 days PSS data, the reactors were operated without the tubes in the settlers zone at 2 h and 1 h to

evaluate the role of tube settlers in the reactor performance. This phase of the study with domestic wastewater lasted for about 125 days.

The COD removal efficiency was expressed in three ways: based on (1) total influent and total effluent (TI-TE); (2) filtered influent and filtered effluent (FI-FE); and (3) total influent and filtered effluent (TI-FE), hereafter referred as COD (TI-FE). The first reflects the organic matter removal by physical action and biological decomposition. The second represents the biological activity. The third represents a treatment potential which could be attained by removing all the suspended solids from the effluent, that is, by employing an ideal sedimentation system. The data obtained on important process parameters during the entire period of operation are presented in Figures 6.14-6.17.

### 6.6.1 Secondary Start-up

The reactors $R_1$ and $R_2$ already containing granular sludge which were earlier used to treat CERELAC-based synthetic wastewater were operated at 3 h HRT. During the initial four days of secondary start-up, half of the feed was made up with raw sewage, the rest being synthetic CERELAC wastewater. Thereafter, for 3 days, the sewage fraction was increased to 75% and from day 8, the feed contained only sewage. On day 9, the HRT of $R_2$ was reduced to 2 h.

The variation of total and soluble COD in both influent and effluent for $R_1$ are given in Figures 6.14a and 6.14b. These parameters for $R_2$ are shown in Figures 6.16a and 6.16b. From day 8 to 23 the total

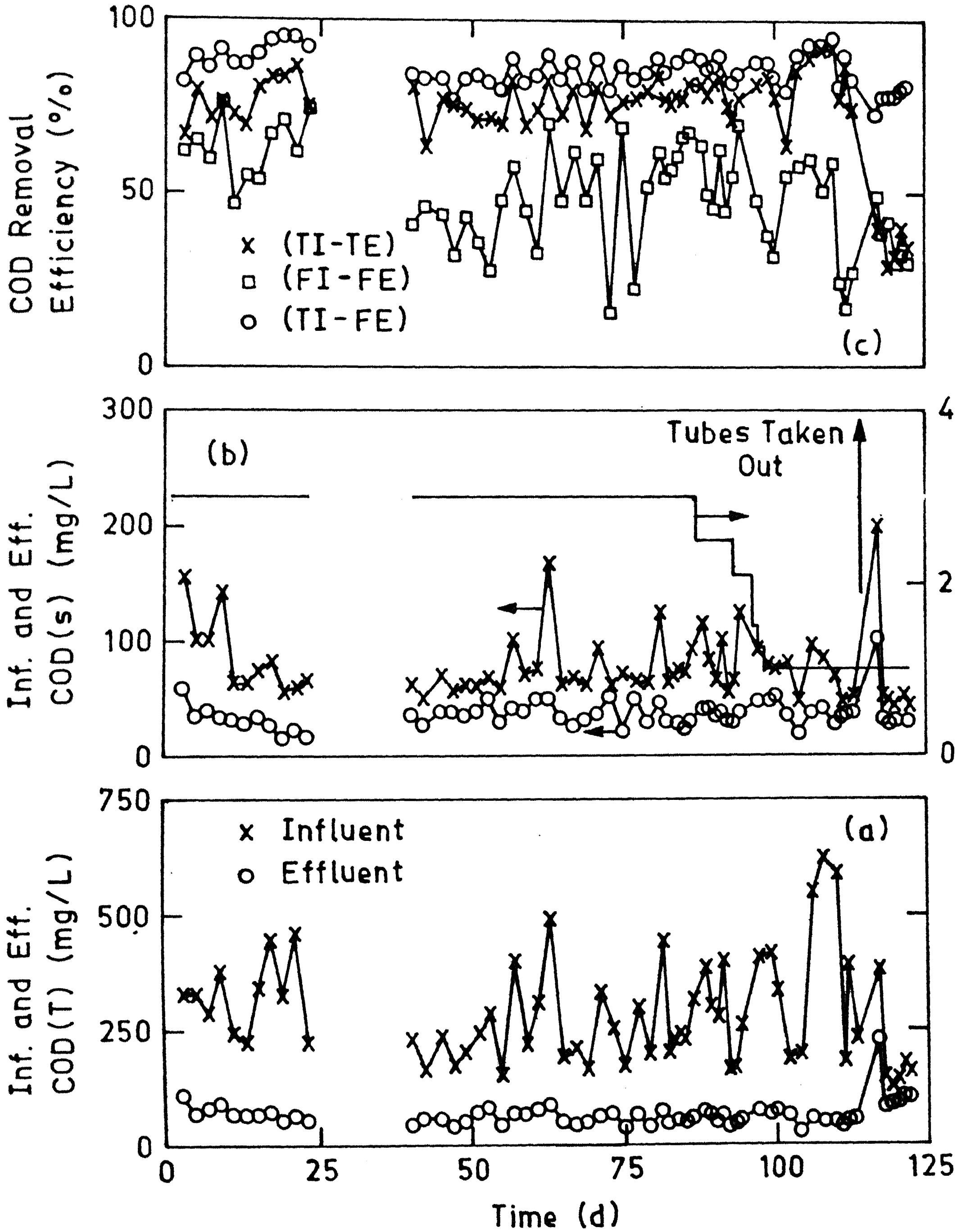



Fig. 6.14. Performance of $R_1$ Fed with Domestic Wastewater.

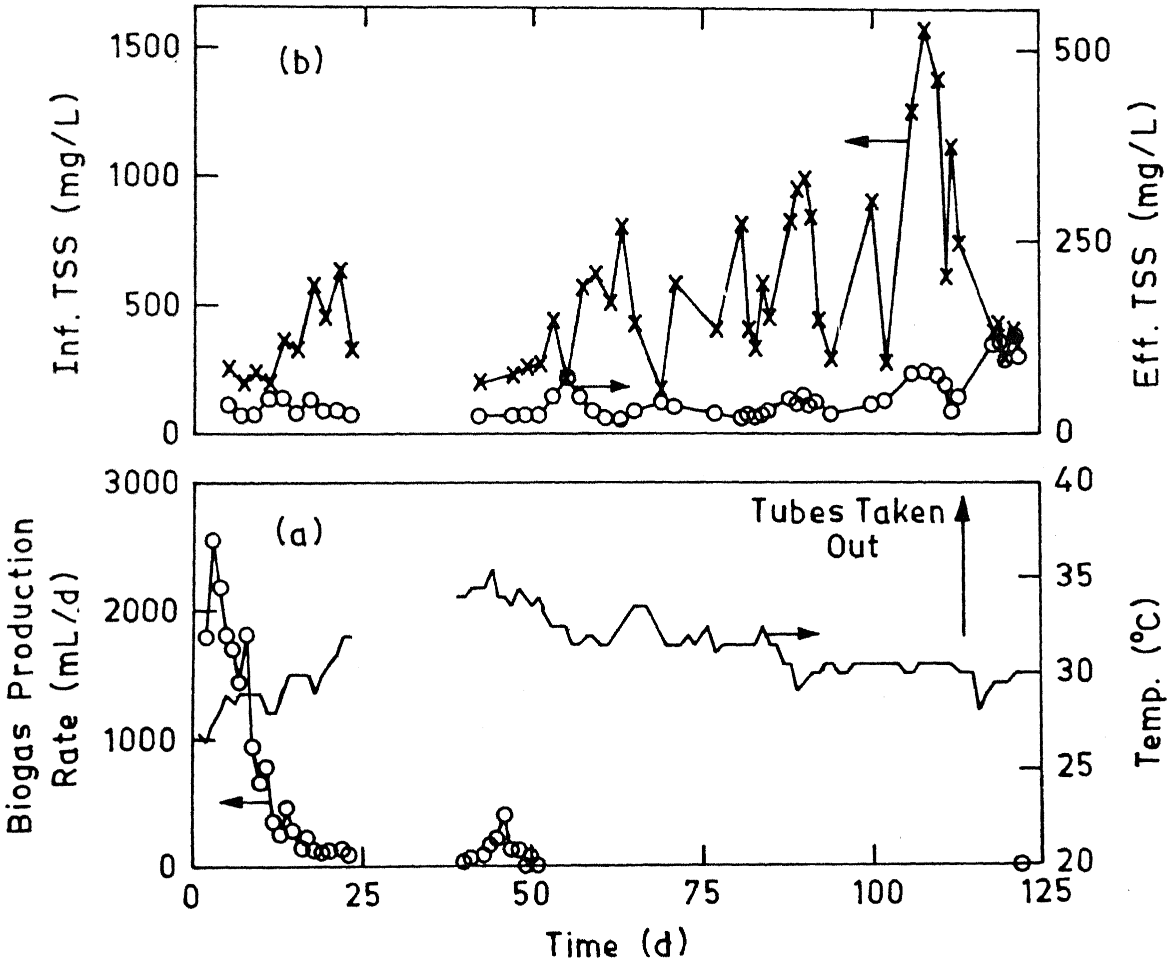



Fig. 6.15. Performance of $R_1$ Fed with Domestic Wastewater.

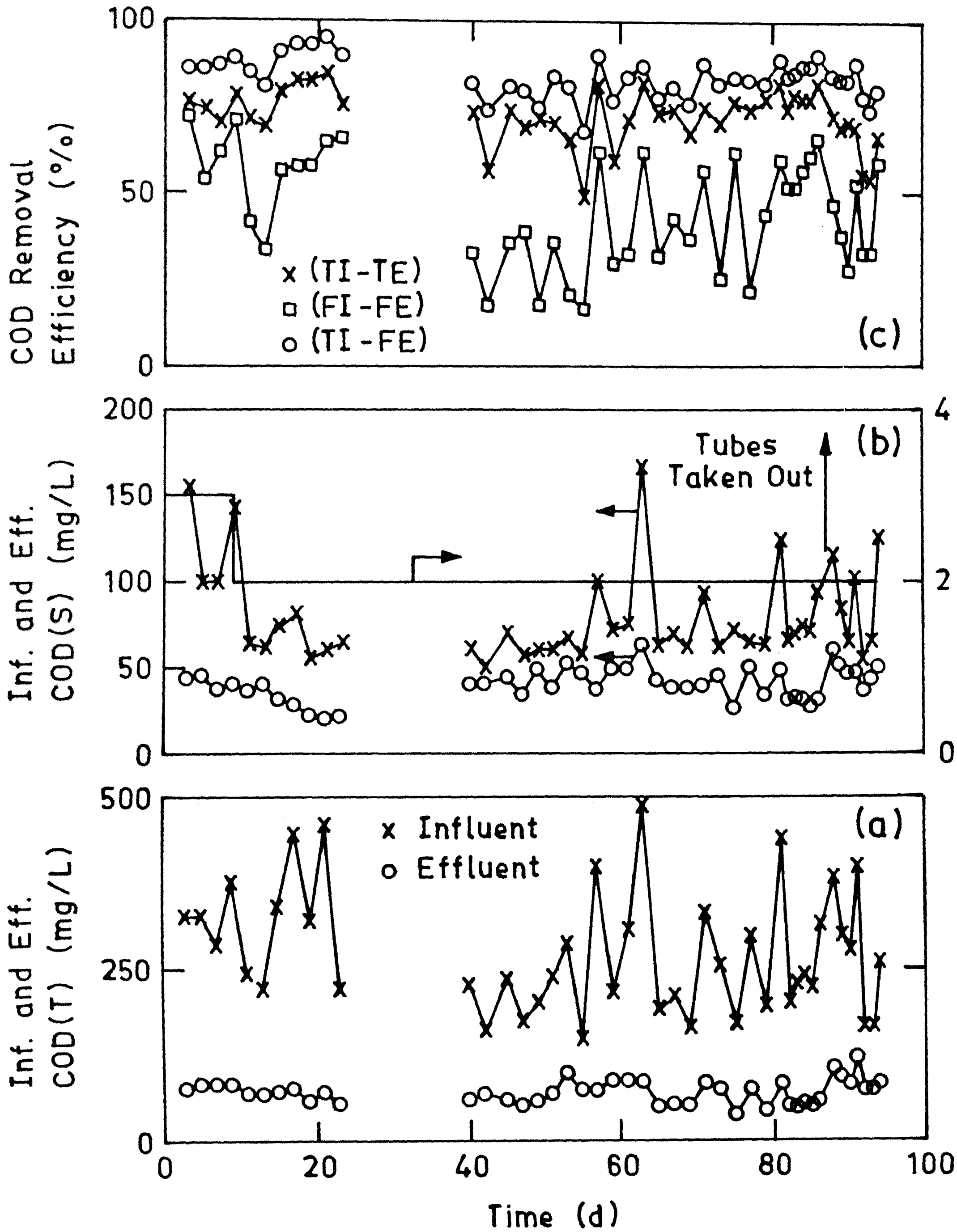



Fig. 6.16. Performance of $R_2$ Fed with Domestic Wastewater.

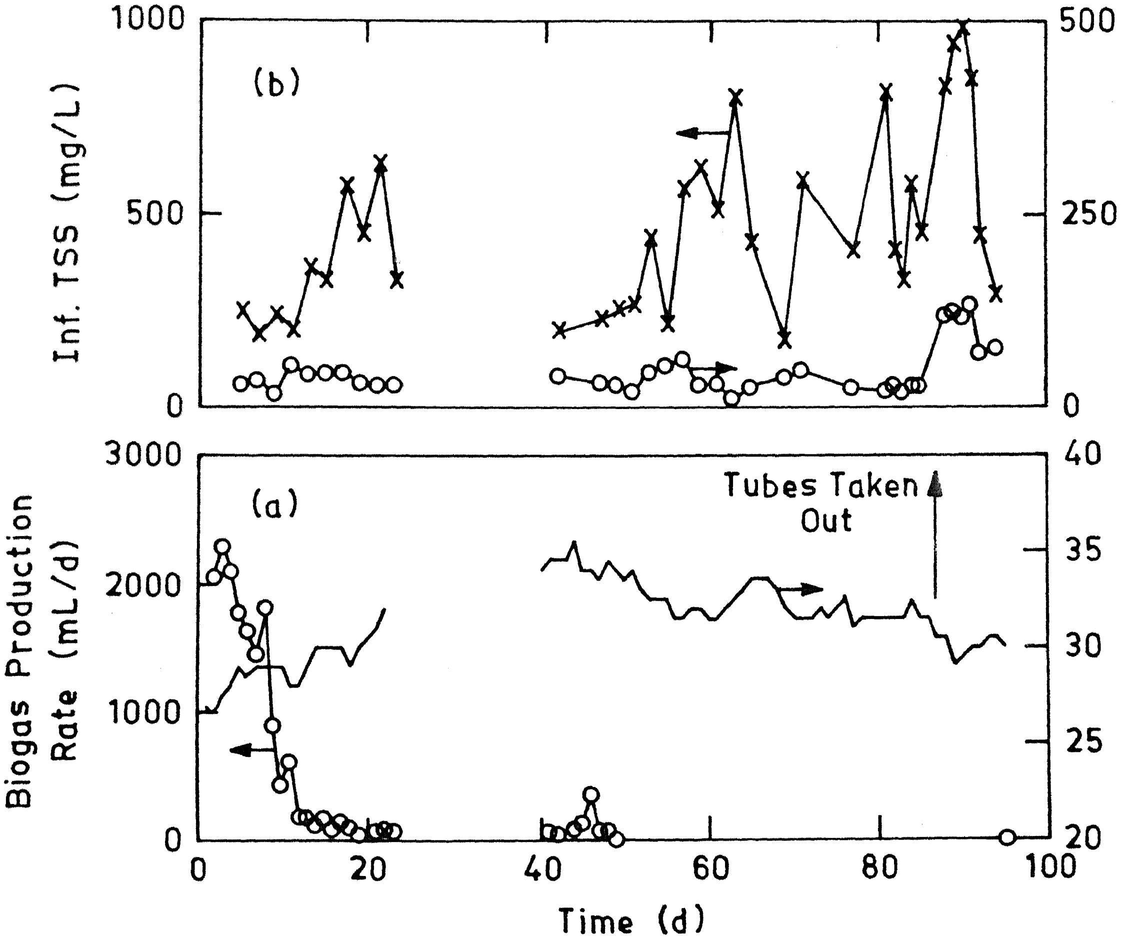



Fig. 6.17. Performance of $R_2$ Fed with Domestic Wastewater.

and soluble COD of the influent sewage to both the reactors were in the range of 220-457 mg/L and 55-145 mg/L respectively.

During these days the total COD removal efficiency (TI-TE) was in the range of 69-85% in both the reactors (Figures 6.14c and 6.16c). The soluble COD removal efficiencies (FI-FE) in $R_1$ and $R_2$ were in the range of 47-74% and 34-71% respectively. The influent pH varied in the range of 7.4-8.2 while the effluent pH in both the reactors were in the range of 7.5 to 7.9. Figures 6.15a and 6.17a shows the variation of gas recovery rate in reactors $R_1$ and $R_2$ respectively. A gradual reduction in gas recovery was observed in both the reactors and from day 15 it remained below 200 mL/d. The TSS content in the influent was in the range of 200-629 mg/L while the effluent TSS was below 50 mg/L in both the reactors (Figures 6.15b and 6.17b).

During the initial 23 days of operation, in general, a decreasing trend in the influent soluble COD was observed. This could be attributed to the gradual reduction in CERELAC content in the wastewater, as the soluble fraction of COD in CERELAC wastewater was 43% whereas in raw domestic wastewater it was only about 24%. The gradual decrease in gas recovery as the reactors were operated solely on raw domestic wastewater indicated the low energy recovery potential of high rate reactors working on low-strength wastewater. In this case, the soluble methane loss in the effluent is significant compared to the methane produced (Kobayashi et al., 1983; Lettinga, 1992).

Figures 6.14c and 6.16c shows the variation of COD removal efficiency. An initial decreasing trend in soluble COD removal (FI-FE)

in both the reactors with the introduction of sewage can be noted. However, the soluble COD removal efficiency gradually improved and a maximum of 74% and 66% in $R_1$ and $R_2$ respectively, were observed on day 23 indicating an improvement in biological activity in the reactors. Concomitant to this the COD (TI-FE) removal efficiency also improved and remained above 90% in both the reactors from day 17 to 23. These observations indicated a reasonable adaptation of the granular sludge to the raw sewage.

### 6.6.2 Reactor Operation at Various HRTs

Feeding of both the reactors were temporarily terminated on day 23 due to serious power failure in the campus. However, after 16 days the reactors were restarted. For few days after this restarting, the COD removal efficiency slightly decreased in both the reactors. After about 16 days of operation the reactor performance improved in general. The operation of $R_1$ and $R_2$ at 3 h and 2 h HRT was continued till day 87 and 94 respectively. Wide fluctuations in influent COD and TSS concentration were observed during these days. However, the effluent quality was less influenced by these fluctuations and the COD and TSS concentrations in the effluent remained fairly constant. The gas recovery from both the reactors was very small after the restart also and it remained zero since day 51 and 48 for $R_1$ and $R_2$ respectively (Figures.6.15a and 6.17a). Steady-state was assumed in both the reactors from day 81. Results of studies conducted for 5 consecutive days to evaluate the steady-state performance of both the reactors are presented later.

From day 87 the HRT of $R_1$ was gradually reduced from 3 h achieving 1 h HRT on day 98. The reactor was operated at this HRT till the end of the study. PSS data pertaining to 1 h HRT was collected from day 108 to day 113.

After completion of PSS data collection at 1 h and 2 h HRTs in $R_1$ and $R_2$ respectively the reactors were opened up and all the tubes were removed from the settler zone. It was observed that all the tubes were free from any chocking. The reactor operation was continued at same HRTs for about 10 more days and the reactor performance was evaluated. During this period the effluent from both the reactors exhibited high TSS concentration (Figures 6.15b and 6.17b). Concomitantly considerable increase in effluent total COD and reduction in COD removal efficiency was observed (Figure 6.14c and 6.16c).

### 6.6.3 Reactor Performance

During the entire period of reactor operation at various HRTs in spite of the considerable fluctuation in the influent characteristics, the effluent COD and TSS concentration remained fairly constant (Figures 6.14 to 6.17). From day 11 to 86 when $R_1$ was operating at 3 h HRT, the influent total COD was in the range of 148-488 mg/L (average 266 mg/L), whereas the effluent total COD was in the range of 40-89 mg/L (average 61 mg/L). This corresponded to total COD removal efficiency of 77%. It may be noted that during this period the termination of feeding for 16 days and restarting of reactor had occurred. From day 87 to 100, during which the HRT of $R_1$ was gradually changed from 3 h to 1 h, the effluent total COD was in the range of 41-

77 mg/L (average 63 mg/L) against an influent COD range of 167-414 mg/L (average 310 mg/L) effecting a treatment efficiency of 80%. During reactor operation at 1 h HRT the influent COD varied between 182 and 622 mg/L whereas the effluent COD varied between 30 and 66 mg/L. Similar removal efficiency was observed in $R_2$ also. This indicated that the reactor configuration was effective in keeping the effluent quality fairly constant, in spite of the wide fluctuations in influent parameters.

Details of reactor performance during PSS at HRTs of 3 h, 2 h and 1 h as well as at 2 h and 1 h HRT without tubes are summarised in Table 6.4.

#### 6.6.3.1 COD and BOD Removal Efficiencies

The average influent total COD during PSS at 3 h, 2 h and 1 h HRT were 266, 266 and 402 mg/L. The influent to $R_1$ and $R_2$ when operating simultaneously at 3 h and 2 h HRT respectively were the same. The average effluent total COD at these HRTs were 57, 59 and 52 mg/L. At 1 h HRT, in spite of the significantly higher influent COD, the effluent quality was not affected. When $R_2$ was operated at 2 h HRT without tubes, the effluent total COD was 96 mg/L for an influent total COD of 305 mg/L . The corresponding values for $R_1$ when operating at 1 h HRT without tubes were 96 mg/L and 149 mg/L (Table 6.4). The higher effluent total COD at 2 h and 1 h HRT, without tube settler compared to that with tube settler, demonstrated the usefulness of such type of arrangement in the settler zone. It may be noted that when $R_1$ was

Table 6.4 Performance of $R_1$ and $R_2$ Fed with Domestic Wastewater at Various HRTs

| Parameter | | | HRT, h | | | | |
|---|---|---|---|---|---|---|---|
| Name | | Unit/Data type | With tubes | | | Without tubes | |
| | | | 3($R_1$) | 2($R_2$) | 1($R_1$) | 2($R_2$) | 1($R_1$) |
| Influent COD | Total | mg/L | 266±122 | 266±122 | 402±250 | 305±115 | 149±25 |
| | Soluble | Average ± $\sigma$ | 81±30 | 81±30 | 59±22 | 84±31 | 48±5 |
| Space loading rate | | g COD/L.d | 2.13 | 3.19 | 9.65 | 3.66 | 3.58 |
| Reactor sludge VSS concentration | | g/L | 3.16 | 2.17 | 6.23 | - | - |
| VSS/TSS ratio | | | 0.41 | 0.36 | 0.23 | - | - |
| Sludge loading rate | | g COD/ g VSS.d | 0.67 | 1.47 | 1.55 | - | - |
| Effluent COD (Efficiency) | Port No. 1 Soluble | mg/L Average ± $\sigma$ ( % ) | 32±11 (60) | 35±10 (57) | 36±7 (39) | 49±11 (42) | 31±3 (35) |
| | Port No. 1 Total | | 57±14 (79) | 59±18 (78) | 52±8 (87) | 96±23 (69) | 96±13 (36) |
| Efficiency | Port No. 1 COD (TI-FE) | % | 88 | 87 | 91 | 84 | 79 |
| Closeness to ideal system in total COD removal | | % | 90 | 90 | 96 | 82 | 46 |
| Infl. $BOD_5$ | Soluble | mg/L | 22 | 22 | 23 | - | - |
| | Total | | 78 | 78 | 114 | - | - |
| Effl. $BOD_5$ | Soluble | | 6 | 7 | 8 | - | - |
| | Total | | 15 | 15 | 14 | - | - |

contd...

Table 6.4 (continued)

| Parameter | | HRT, h | | | | |
|---|---|---|---|---|---|---|
| Name | Unit/Data type | With tubes | | | Without tubes | |
| | | 3($R_1$) | 2($R_2$) | 1($R_1$) | 2($R_2$) | 1($R_1$) |
| Space substrate removal rate | g COD/L.d | 1.68 | 2.49 | 8.4 | 2.53 | 1.29 |
| Specific substrate removal rate | g COD/ g VSS.d | 0.53 | 1.15 | 1.35 | - | - |
| Methane recovery rate-NTP | mL/L.d Average ± σ | 0 | 0 | 0 | 0 | 0 |
| Influent TSS | mg/L Average ± σ | 518±236 | 518±236 | 1086±511 | 812±265 | 373±66 |
| Influent VSS | | 179±73 | 179±73 | 314±184 | 247±67 | 130±32 |
| Effluent TSS | | 24±5 | 25±6 | 60±27 | 114±31 | 111±16 |
| Effluent VSS | | 17±4 | 17±5 | 43±17 | 55±18 | 54±11 |
| Wasted sludge VSS | mg/L eff | 6 | 5 | 8 | - | - |
| VSS/TSS ratio | | 0.46 | 0.42 | 0.24 | - | - |
| BSRT | d | 17 | 8 | 5 | - | - |
| Specific methanogenic activity | g $CH_4$-COD/ g VSS.d | 0.17 | 0.20 | 0.12 | - | - |
| Temperature | $^0C$ | 32 | 32 | 30 | 30 | 30 |

operating at 1 h HRT without tubes the influent COD was significantly lower, still, the effluent COD was higher than that with tubes.

Figure 6.18 shows the variation of total COD and COD (TI-FE) removal efficiency when the reactors were operated at different HRTs with and without tubes in the settler zone. The total COD removal efficiencies at 3 h, 2 h and 1 h HRT were 79%, 78% and 87% respectively. The corresponding COD (TI-FE) removal efficiencies were 88%, 87% and 91%. These are the once which can be achieved with an ideal settler system. This means that, the reactor configuration used in the study was close to the extent of 90%, 90% and 96% to the ideal system at 3 h, 2 h and 1 h HRT respectively. The observed higher effectiveness of tubes at 1 h HRT has resulted from lower fraction of suspended COD in the effluent in spite of the higher suspended COD in the influent.

When $R_2$ was operated at 2 h HRT without tubes, the total COD and COD (TI-FE) removal efficiencies were 69% and 84% respectively. Corresponding values at similar situation at 1 h HRT were 36% and 79%. This indicated that the role of tube settlers is more prominent at 1 h HRT.

The influent and effluent $BOD_5$ when the reactors were operating at various HRTs are given in Table 6.4. The average total $BOD_5$ of the effluent at 3 h and 2 h HRT was 15 mg/L and at 1 h HRT it was 14 mg/L. The soluble effluent $BOD_5$ at this HRTs were in the range of 6-8 mg/L.

#### 6.6.3.2 TSS and VSS Removal

The TSS concentration of the domestic wastewater varied over a wide range (171 - 1575 mg/L). However, the effluent TSS

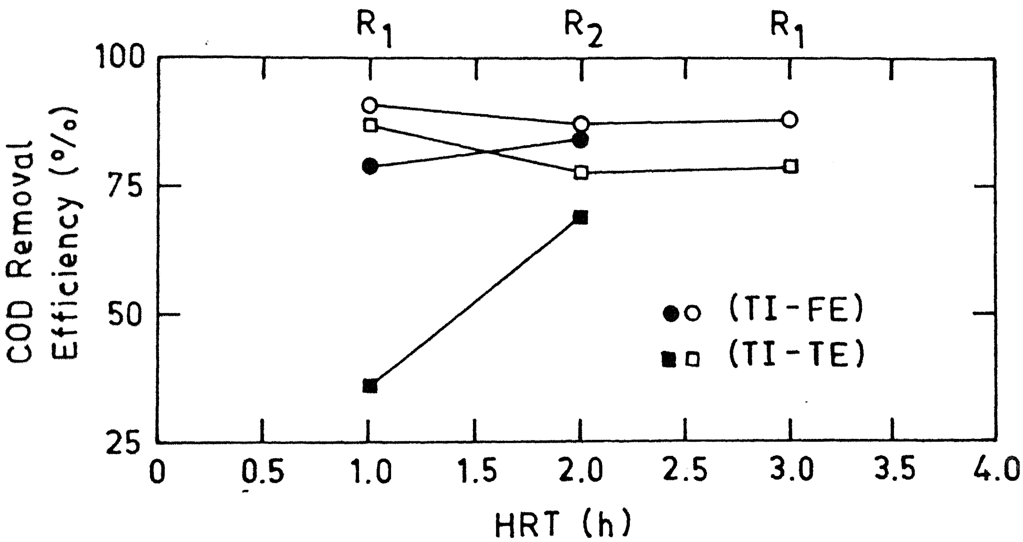



Fig. 6.18. Steady State Reacter Performance in Terms of COD Removal for Domestic Wastewater. (○ and □) with Tubes (● and ■) without Tubes.

concentrations were fairly stable (Figures 6.15b and 6.17b). The TSS and VSS concentration of the influent and effluent corresponding to various HRTs are given in Table 6.4. The average TSS and VSS of the wastewater used at 3 h as well as 2 h HRT was 518 mg/L and 179 mg/L respectively. At 1 h HRT these values were 1086 mg/L and 314 mg/L. The effluent TSS at 3 h, 2 h and 1 h HRT were 24, 25 and 60 mg/L. This corresponds to a TSS removal efficiency in the range of 94-95%. However, when the reactors were operated without tubes the effluent TSS were 114 and 111 mg/L at 2 h and 1 h HRT which corresponded to a removal efficiency of 86% and 70%. This demonstrated that the tubes were effective in achieving high suspended solids removal efficiency.

#### 6.6.3.3. Sludge Accumulation and Wasting

The VSS concentration in $R_1$ and $R_2$ were 3.16 and 2.17 g/L respectively at PSS corresponding to HRTs of 3 h and 2 h. It may be recalled that just prior to the introduction of sewage to the two reactors, that is when $R_1$ and $R_2$ were at 2 h and 1 h HRT on CERELAC wastewater, the VSS concentration were 7.0 and 6.14 g/L. In spite of the comparatively low VSS concentration in the reactor, when operated with domestic wastewater, regular sludge withdrawal through port No.2 had to be continued to maintain the sludge level at 50 cm height. This indicated the loosely packed nature of the sludge bed at the relatively low loading rate of 2.13 and 3.19 g COD/L.d in $R_1$ and $R_2$ respectively, possibly due to holding of gas in the sludge bed itself. As the HRT of $R_1$ was reduced to 1 h, the space loading rate increased to 9.65 g COD/L.d corresponding to an influent COD of 402 mg/L with 85% of

insoluble fraction. At this HRT the reactor VSS increased to 6.23 g/L. This increase in VSS concentration indicated the accumulation of suspended solids in the reactor at the increased loading rate.

Further, as the HRT decreased from 3 h to 1 h, the reactor TSS increased from 7.73 g/L to 27.67 g/L and the sludge VSS/TSS ratio decreased from 0.41 to 0.23. This increased inorganic content in the sludge might have resulted in a reduced sludge bed expansion leading to the formation of a densely packed sludge bed. It may be noted that the VSS/TSS ratio of solids in the influent at 1 h HRT was 0.29. This indicated that the reactor VSS concentration and the VSS/TSS ratio are greatly influenced by the concentration and nature of suspended solids in the influent.

The VSS and TSS concentrations of 6.23 and 27.67 g/L observed in the reactor at 1 h HRT are well comparable with those reported in the literature. Lettinga (1992) has reported a VSS and TSS concentration of 9.4-12.5 and 31-37.5 g/L in full scale UASB reactors treating domestic wastewater. The higher ratio of reaction zone volume/total reactor volume have to be considered here. In full scale units about 60% of the reactor volume is reaction zone which is occupied by the sludge; this is based on a reactor height of 4 m (Lettinga, 1992) and a gas-liquid-solid separator height of 1.5 m (Lettinga and Hulshoff, 1992). In the present investigation only 40% of the reactor volume was occupied by the sludge and thus the sludge concentration in the reactors was comparable with that of full scale units.

The average excess sludge production (including sludge in the effluent and that wasted) with respect to the influent total COD was calculated to be 0.14 g COD/g $COD_{in}$ for the range of HRTs employed. According to Lettinga (1992), the excess sludge production in full-scale UASB reactors is generally 0.1 g COD/g $COD_{in}$. The excess sludge production in terms of TSS with respect to the influent TSS concentration during the PSS at various HRTs was in the range of 7.1 X $10^{-2}$ - 8.6 X $10^{-2}$ g TSS/g $TSS_{in}$. This is significantly low compared to an excess sludge production rate of 0.4-0.6 g TSS/g $TSS_{in}$ reported by Lettinga (1992). The relatively low excess TSS production indicated accumulation of suspended inorganic compounds of the influent TSS in the reactor. It may be recalled that the VSS/TSS ratio was lower at 1 h HRT. However, operation of the reactor for a longer period is needed to get a better assessment of the excess sludge production.

#### 6.6.3.4 Substrate Utilisation Rate

In reactor $R_1$ for a space loading rate of 2.13 g COD/L.d (at 3 h HRT) the space substrate utilisation rate was 1.68 g COD/L.d. As the loading rate increased to 9.65 g COD/L.d at 1 h HRT, the space substrate utilisation rate increased to 8.4 g COD/L.d. In $R_2$ at 2 h HRT, the space substrate utilisation rate was 2.49 g COD/L.d corresponding to a loading rate of 3.19 g COD/L.d. As the specific loading rate increased from 0.67 to 1.55 g COD/g VSS.d, the specific substrate utilisation rate increased from 0.53 to 1.35 g COD/g VSS.d. These observations show the high COD removal capacity of these reactors even at an HRT of 1 h.

Though the total COD removal efficiency increased from 79% to 87% as the HRT reduced from 3 h to 1 h the soluble COD removal efficiency has reduced from 60% to 39% indicating a decrease in the biological activity in the reactor.

#### 6.6.3.5 Methane Production

During the continuous operation of reactors on domestic wastewater no gas could be recovered. It appears that the methane formed in the reactor was less than what could be dissolved, which may be even more than the equilibrium concentration, and lost in the effluent. Moreover, the insoluble fraction of COD being high (70-85%), it appeared that the COD removal was mostly due to the entrapment of suspended organic solids in the sludge bed contributing significantly to the VSS increase in the reactor. The overall bacterial activity was comparatively low as indicated by the reduced soluble COD removal efficiency. At the extremely low HRTs employed in this investigation the complete liquefaction and biomethanation of suspended organic matter appeared to be impaired affecting the gas production rate. Similar observations of reduced biodegradation rate of suspended organic mater directly affecting gas production were made by Barbosa and Sant'Anna Jr. (1989) while treating domestic wastewater of high solid content at 4 h HRT in conventional UASB reactor.

After completion of the performance evaluation of $R_1$ at 1 h HRT without tubes, the feeding was terminated. However, the gas measurement system was kept connected to the reactor. During the following ten days, 2090 mL of total biogas was recovered from the reactor. A maximum

gas of 370 mL was recovered on day 5 after termination of feeding. This gas has resulted from the degradation of the already accumulated suspended organic matter in the reactor.

The SMA was in the range of 0.2 to 0.12 g $CH_4$-COD/g VSS.d. This activity was comparable with the value reported in literature. Lettinga (1992) reports an SMA >0.1 g $CH_4$-COD/g VSS.d for UASB sludge. It may be noted that as the HRT was decreased from 3 h to 1 h in $R_1$ the SMA reduced from 0.17 to 0.12 g $CH_4$-COD/g VSS.d. At 1 h HRT, the suspended fraction of sewage was 85% effecting the reactor VSS increase to 6.23 g/L from 3.16 g/L. The lower SMA at 1 h HRT indicates a low methanogenic population in the reactor, possibly due to the accumulation of suspended organic matter and reduced availability of acetate.

### 6.6.4 Settling Characteristics of the Reactor Sludge

Settling analysis of sludge from $R_1$ and $R_2$ was conducted using a glass column of 3.5 cm diameter and 192 cm height based on the method suggested by Sanchez Riera et al. (1985). Equal quantities of sludge drawn from the bottom four ports at the end of reactor operation at 1 h and 2 h HRT were mixed and used for this study. For comparison, the sludge from 5 MLD UASB reactors, Kanpur (India) drawn from the one fourth and three fourth height of sludge bed in October 1995, and mixed, were also subjected to similar study. The settling column was filled with water and 50 mL of the sludge sample was added quickly from the top. TSS and VSS concentration of the samples drawn at different time interval from bottom of the column, ensuring the complete removal of settled portion were analysed.

The terminal velocity of each group of particles was calculated as the ratio between the average water column length and sampling time. The TSS percentage of the settled solids referred to the total solids that settled faster than 0.03 m/min was calculated and were used for the subsequent analysis. The cumulative velocity distribution curves were obtained for the three samples by plotting the TSS percent of the solids that settle slower than the corresponding settling velocity as a function of the settling velocity. This curve was used to prepare histogram showing the settling velocity distribution for the three sludges (Figure 6.19). This figure shows that 29% and 27% of the sludge particles in $R_1$ and $R_2$ respectively, had settling velocities greater than 0.9 m/min (54 m/h), a value greater than 0.83 m/min (50 m/h) which is reported to be typical for granular sludge (Stronach et al., 1986).

About 58% of the sludge from $R_1$ and $R_2$ had a settling velocity of 0.1 to 0.3 m/min indicating that a considerable fraction of the sludge was not in granular form at the end of reactor operation at 1 h and 2 h HRT with domestic wastewater. A comparison of photographs of diluted sludge from port No. 5 (Figure 6.20a) and from port No. 2 (Figure 6.20b) of $R_1$ at the end of reactor operation, indicated that the bottom portion of the sludge zone contained less granules. This might have resulted from the displacement of granular sludge from bottom of the sludge zone to the top by the suspended solids in the influent.

In the case of sludge from 5 MLD UASB treating domestic wastewater all the particles had settling velocities much lower than 0.9

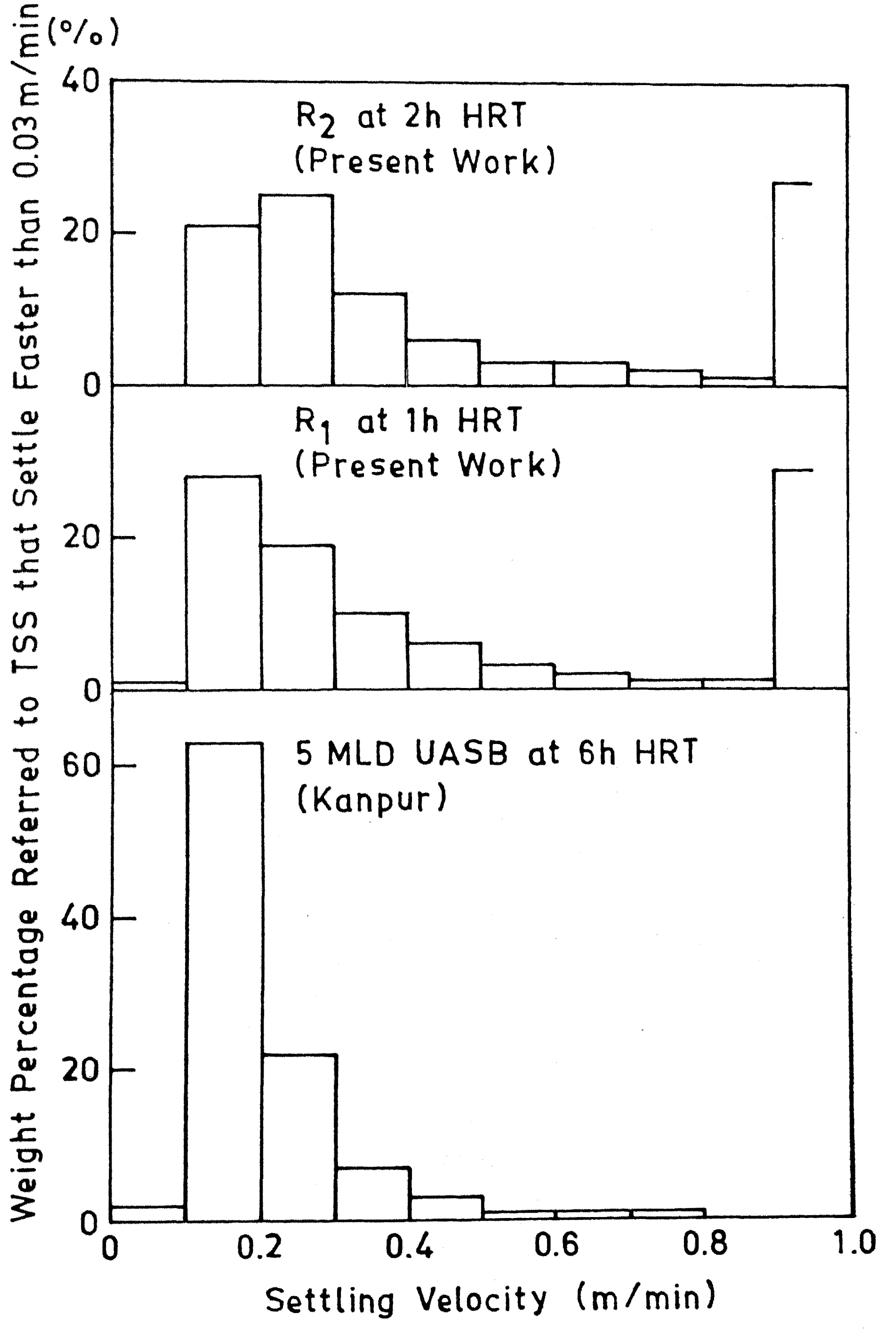
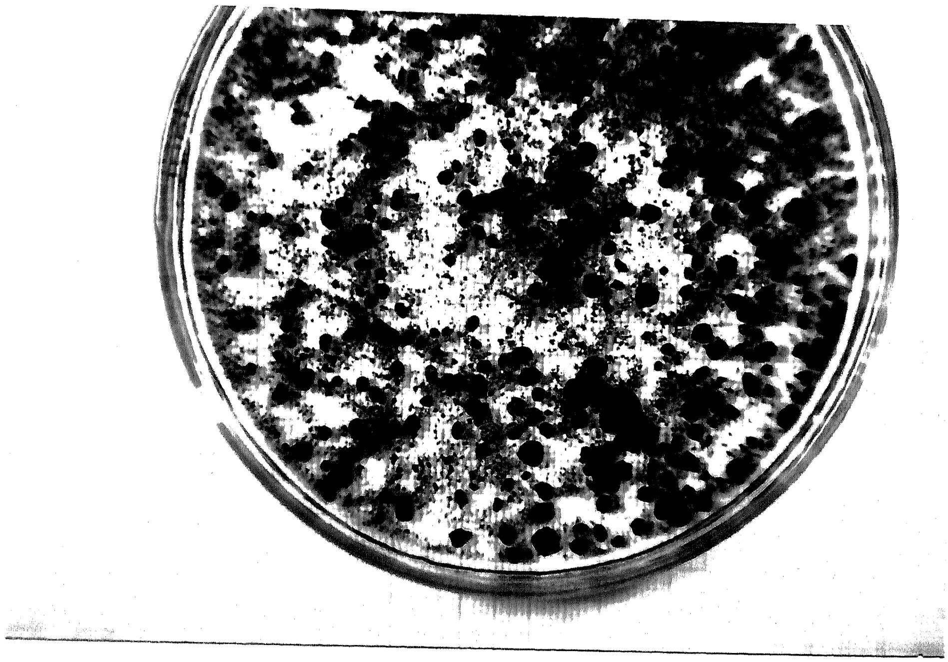
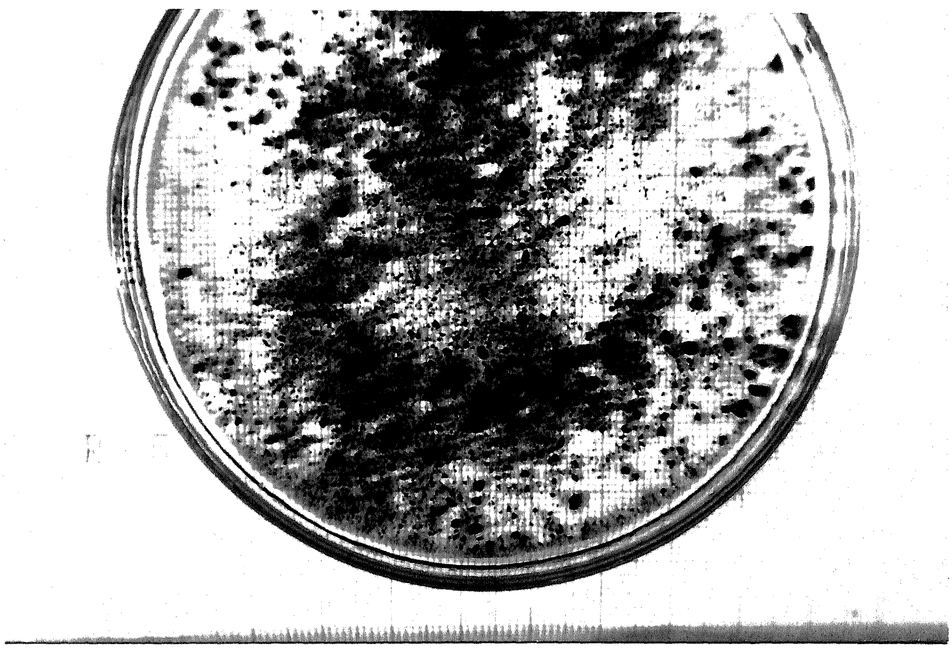



Fig. 6.19. Settling Velocity Distribution of Anaerobic Sludge from Various Reactors Fed with Domestic Wastewater.



(b) Sludge from Port No. 2



(a) Sludge from Port No. 5

Figure 6.20 Photograph of Diluted Sludge Samples from $R_1$ Fed with Domestic Wastewater at 1 h HRT.

m/min. However, 92% of the sludge was having a settling velocity of 0.1 to 0.3 m/min.

## 6.7 Effect of Type of Wastewater on Reactor Performance

One of the objectives of this investigation was to compare the suitability of the modified UASB reactor configuration in treating wastewaters of simple and complex nature. A comparison of the reactor performance while treating synthetic wastewater based on sucrose and CERELAC and raw domestic wastewater is presented in the following section.

### 6.7.1 Response of Reactor Sludge Bed to Different Types of Wastewater

During the steady-state operation at various HRTs with different types of wastewaters, studies were conducted to evaluate the reactor performance along the height of sludge bed. The sludge samples drawn from the bottom four ports were used to evaluate various performance parameters. Representative data obtained from this study for different substrates are presented in Figures 6.21 and 6.22.

Figure 6.21(a)-(d) shows the variation of soluble COD along the sludge height of $R_2$ at 2 h HRT while treating the three types of wastewater and of $R_1$ at 1 h HRT while treating domestic wastewater. A general trend in reduction of soluble COD along the height was observed. On comparison of Figure 6.21 (a) and (b) it can be seen that in case of completely soluble substrate, most of the waste stabilisation occurred at the bottom half of the sludge bed. In the case of CERELAC wastewater, the soluble COD remained above 290 mg/L up to port No. 3

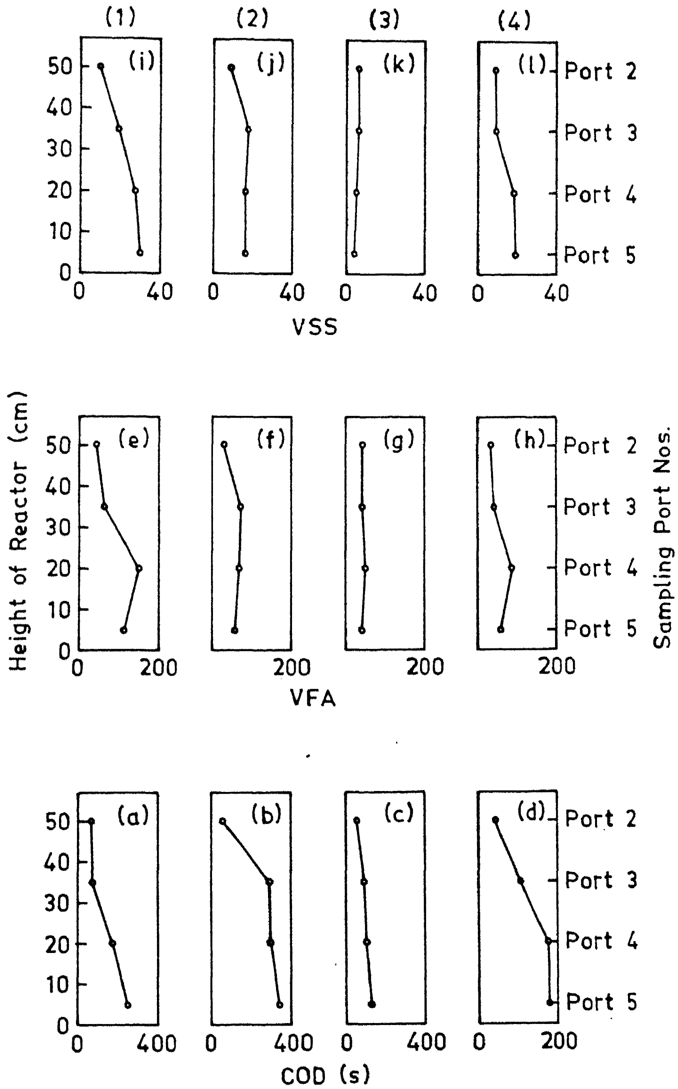



Fig. 6.21. Profile of COD(s), VFA and VSS.
(1) Sucrose-Based Wastewater - $R_2$ at 2h
(2) Cerelac-Based Wastewater - $R_2$ at 2h
(3) Domestic Wastewater - $R_2$ at 2h
(4) Domestic Wastewater - $R_1$ at 1h

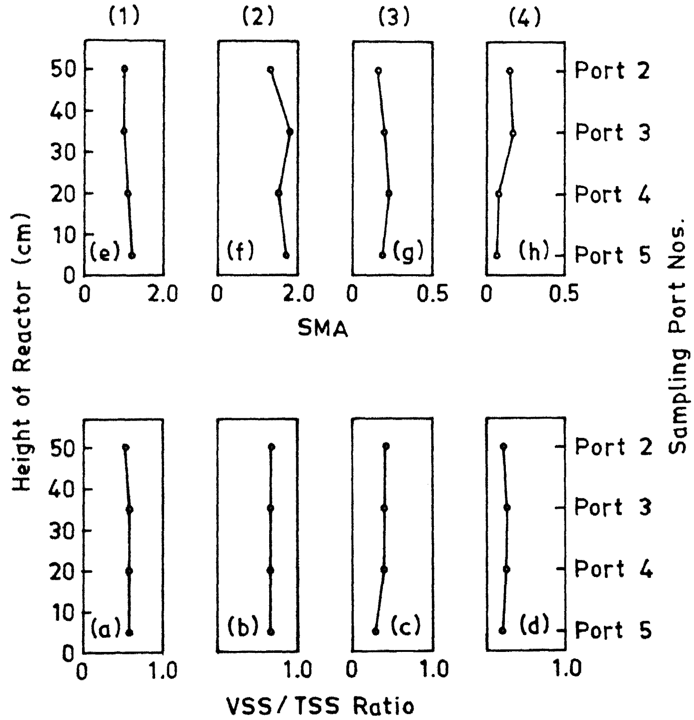



Fig. 6.22. Profile of VSS/TSS Ratio and SMA.
(1) Sucrose-Based Wastewater - $R_2$ at 2h
(2) Cerelac-Based Wastewater - $R_2$ at 2h
(3) Domestic Wastewater - $R_2$ at 2h
(4) Domestic Wastewater - $R_1$ at 1h

whereas the influent soluble COD was about 200 mg/L. This indicated that the hydrolysis of suspended organic matter was prominent over two third height of the sludge zone. Methane formation from acid was mostly concentrated between port No. 2 and 3. When $R_2$ was at 2 h HRT with domestic wastewater (Figure 6.21c), the soluble COD remained more or less constant and this may be attributed to the low COD of the influent. At 1 h HRT (Figure 6.21d) when the influent COD was comparatively high, a significant decrease in soluble COD was only observed between ports No. 4 and 2.

Figure 6.21(e)-(h) shows the variation of VFA along the sludge bed height. In the case of sucrose wastewater an accumulation of VFA between ports No. 5 and 4 can be observed indicating a higher rate of acidification compared to methane formation in this zone. Similar situation can be seen in Figure 6.21(h) when the reactor was operated at 1 h HRT on domestic wastewater containing high suspended fraction of COD. Figure 6.21(f) and (g) indicate that in the case of CERELAC wastewater and domestic wastewater of low-strength there was a balance between acid production and acid degradation. A comparison of soluble COD profile with VFA profile shows that the sludge zone contains short-chain and long-chain fatty acids for which the COD/VFA ratio is higher than that of acetic acid. In the case of CERELAC wastewater and domestic wastewater of high strength, this situation was more prominent possibly due to the presence of fat in the wastewater. Presence of non-acidified soluble organic matter like glycerol and amino acids also can result in this type of situation.

The observations presented thus far indicated that in the case of wastewater containing insoluble organic matter, hydrolysis and acid formation are prominent over a higher percentage of the sludge bed height. It may be also inferred that the sludge zone functions more like a plug flow reactor with minimum vertical mixing.

Figure 6.21(i)-(l) and Figure 6.22(a)-(d) show the profile of VSS and VSS/TSS ratio respectively along the sludge bed height for various wastewater feeding. It can be observed that, the reactor VSS content and VSS/TSS ratio were, in general, relatively higher when the reactors were on sucrose or CERELAC wastewater compared to domestic wastewater. A higher rate of cell synthesis on these substrate and very low inorganic content in the influent might have resulted in this high sludge concentration. Moreover, the relatively higher loading rate and resulting gas production can lead to an improved bed agitation and thickening of the sludge bed (Lettinga et al., 1980). A low VSS concentration at the level of port No. 2 could be due to the regular sludge wasting. The relatively higher VSS concentration at bottom of the reactor in the case of sucrose wastewater is indicative of increased biological activity in this zone.

At 1 h HRT in $R_1$, the influent domestic wastewater was having a relatively higher COD with about 85% in suspended form. This resulted in the higher VSS concentration at bottom two ports. However, the low VSS/TSS ratio in the reactor might have resulted from the low VSS/TSS ratio of the solids in the influent domestic wastewater.

Figure 6.22(e)-(h) shows the variation of SMA of sludge at various heights of the sludge bed. Higher methanogenic activity was exhibited by the reactor sludge when it was on sucrose or CERELAC-based wastewater compared to domestic wastewater. This indicated the higher activity of the sludge on these easily biodegradable substrates.

Along the bed height the highest activity of 1.8 g $CH_4$-COD/g VSS.d was observed for the sludge drawn from port No. 3 when the influent was CERELAC-based water (Figure 6.22f). The high SMA exhibited by the sludge could be due to the very high percentage of acetoclastic methanogens in the sludge granules. This indicated that as the CERELAC contained a wide variety of minerals and nutrients, the wastewater based on this substrate, promoted better growth of the fastedious acetoclastic methanogens. A relatively low SMA exhibited by the sludge when the reactor was treating domestic wastewater, indicated the lower percentage of methanogens in the sludge. This might have resulted from the accumulation of suspended organic matter in the sludge bed from the wastewater. The SMA of sludge from the bottom two ports of $R_1$ at 1 h HRT (Figure 6.22h) was relatively lower indicating a higher accumulation of suspended organic matter at the bottom of the reactor. The displacement of granular sludge from bottom of the sludge zone to the top by the suspended solids in the influent is also evident in the photographs of sludge from port No. 5 and 2 (Figure 6.20a and b). However, the sludge at the bottom of reactor contained acid formers as indicated by the VFA accumulation in this zone (Figure 6.21h).

The average methane production from sucrose and CERELAC-based wastewaters was 0.7 and 0.47 g$\cdot$$CH_4$-COD/g COD removed respectively. The low methane yield during CERELAC wastewater feeding indicates that considerable fraction of COD removed might be in the form of precipitated or adsorbed solids or fat. Moreover, fraction of COD removed may also be accounted for the scum formation at the liquid-gas inter-phase. In the case of domestic wastewater, no methane could be recovered from the reactor. The high suspended fraction of influent COD, relatively impaired biodegradation of the accumulated suspended organic matter in the reactor at the extremely low HRTs employed and the loss of methane in dissolved form in the effluent might have contributed to this.

When the reactors were fed with sucrose and CERELAC-based wastewater, the average sludge yield coefficients were 0.22 and 0.23 g VSS-COD/g COD removed. The former value is comparable with the maximum yield coefficient of 0.28 reported for carbohydrate by Young and McCarty (1974). Hulshoff Pol (1989) has reported a sludge yield of 0.13 g VSS-COD/g COD removed during sucrose rich wastewater feeding. This value does not include the biomass lost through effluent. The high sludge yield with CERELAC wastewater can be attributed for the low BSRTs and low reactor temperature for which the microbial decay rate will be low. The fraction of COD removed in the form of precipitated or adsorbed solids or grease also can contribute to the high yield.

The average excess sludge production with respect to the influent total COD was 0.14 g COD/g $COD_{in}$. This is comparable with the value of

0.1 g COD/g $COD_{in}$, reported by Lettinga (1992), for full-scale UASB reactors treating domestic wastewater. The excess sludge production with respect to the influent TSS concentration was in the range of 7.1 X $10^{-2}$ - 8.6 X $10^{-2}$ g TSS/g $TSS_{in}$. The corresponding values reported by Lettinga (1992) for full-scale UASB reactors are 1.4-0.6 g TSS/g $TSS_{in}$. This relatively low excess TSS yield indicated accumulation of the suspended inorganic compounds of the influent TSS in the reactor.

The BSRTs in the reactor for various HRTs were relatively lower. The sludge withdrawal through port No. 2 has significantly contributed to this. Moreover, the percentage of volume of sludge zone in the present investigation was 40 as compared to about 60 in full-scale units. This will lead to a relatively lower total VSS content in the reactor resulting in lower BSRTs.

### 6.7.2 Effect of Type of Wastewater on the Settler Performance

Over the range of loading rates studied the overall performance of the reactors was only marginally influenced by the change in type of wastewater. The usefulness of the provision of tube settlers in the settler zone was clearly evident while treating all the three types of wastewaters. The soluble COD removal in this zone was marginal. The settlers were very effective in removing suspended fraction of the COD. On comparison of total COD removal efficiency based on samples from port No. 1 and 2 for sucrose and CERELAC-based wastewaters, it can be seen that the improvement effected by the tube settlers was more in the case of the later probably because of the higher suspended solids entering the settler at this case.

The insoluble fraction of the effluent COD, in general, was higher during CERELAC wastewater feeding compared to that with sucrose wastewater or domestic wastewater. However, the effluent VSS concentration was less when the reactors were fed with CERELAC wastewater indicating a higher COD/VSS ratio of the suspended solids in this case.

Irrespective of the wastewater used in the study, the effluent TSS concentration was less influenced by the HRT except at 1 h with CERELAC wastewater. The COD and TSS concentration in the effluent remained fairly stable even at low HRTs during domestic wastewater feeding despite the wide fluctuation in the influent.

The bench-scale reactors used in this investigation were 91-95% close to an ideal system while treating sucrose-based wastewater over the range of HRT studied. While treating CERELAC-based wastewater this closeness was about 90% at 4 and 2 h HRTs. In the case of domestic wastewater the reactors were 90-96% close to the ideal system.

### 6.8 Wasting of the Excess Sludge

During this investigation, the excess sludge was wasted from port No. 2 effecting removal of sludge from top of the bed. When the reactors were on CERELAC-based wastewater, the gas production was significant. The agitation caused by the gas would have helped in the accumulation of the lighter flocs as well as unhydrolysed fraction of suspended organic matter from the influent, above the granular sludge. In these situations sludge wasting from the top would help in enriching the sludge bed with granular sludge. However, in the case of domestic

wastewater feeding, the sludge wastage from top was not beneficial. As the gas production was zero, segregation of granules from the influent suspended organic matter and its accumulation at bottom was not effected. On the contrary, the finer particles, probably the organic matter from the influent, had replaced the granular sludge from bottom to the top of the sludge bed (Figure 6.20a and b). Thus, though the reactor contained considerable amount of granular sludge at the start of domestic wastewater feeding, the reactors would have probably become devoid of any granules if the reactor operation were to be continued. At least in this case wastage of excess sludge from bottom of the reactor might have been a better option effecting the removal of non-granular sludge from the reactor. Thus it may not be possible to suggest a unique location from where the sludge wasting can be done for all types of wastewaters. However, assessment of profile of VSS, VSS/TSS ratio and sludge activity can give required information, to decide on the height from where the sludge wasting has to be done.

### 6.9 Evaluation of Settler Design

Close observations during reactor operation revealed that the sludge particles/flocs ejected from the sludge bed due to the pulse-like liberation of the gas entrapped in the sludge were effectively settled in the tubes. Finer sludge particles could be seen moving up with attached gas bubbles above the sludge zone and entering the settler zone. These particles were also removed in the tube settlers after releasing the gas as hardly any sludge particles were seen in the effluent. After completion of the steady-state operation at various

HRTs with each type of wastewater, the reactors were opened up and on examination of the tubes no chocking was observed in any of the tubes. This indicated that the trapped suspended solids were sliding back to the sludge zone.

Irrespective of the wastewater used in the study, the effluent quality was not much influenced by the HRT, except at 1 h with CERELAC wastewater where the effluent total COD and TSS were 252 mg/L and 201 mg/L respectively. It appears that at this HRT, the settlers were not effective in separating the gas bubbles from the biomass and hence were unable to retain the solids in the reactor. The tube diameter of 2 cm might have become critical in this situation. Because of the insufficient space in the tube, the sludge particles/flocs might have been forced out through the tube by the gas bubbles at this hydraulic loading. Tubes of higher diameter may permit the free passage of gas bubbles without affecting the settling of suspended particles. At 1 h HRT with domestic wastewater the effluent total COD and TSS were 52 mg/L and 60 mg/L. This indicated that at an average flow velocity of 1.24 m/h in the tubes corresponding to 1 h HRT, the particle settling was not affected when the gas loading rate was zero. But at the same flow velocity when the gas loading was 0.04 m/h which was observed with CERELAC wastewater at 1 h HRT, the settling efficiency has considerably reduced. However, for the same wastewater at 2 h HRT, that is for a flow velocity of 0.62 m/h and gas loading of 0.05 m/h, the settler functioning was not affected. This shows that both the flow velocity and the gas loading rate significantly affect the solids settling

efficiency. For tubes with higher diameter the role of gas loading may not be this significant. Thus along with flow velocity in the tube, the gas loading rate also is an important parameter to be considered in design of the settler zone.

Table 6.5 gives values of some important performance parameter at various HRTs with the three types of wastewaters. The high treatment efficiencies exhibited by the reactors in general indicate the usefulness of this type of reactor configuration for the treatment of low-strength wastewater. Only marginal difference in performance was observed between the two reactors $R_1$ and $R_2$ with settler inclination of $45^o$ and $60^o$.

The reactor $R_1$ designed and used in this investigation was later compared with a bench-scale conventional UASB reactor of 8.3 litre liquid volume and 1.12 m height feeding domestic wastewater by Gawande (1996). Severe sludge washout was observed in the UASB reactor at an HRT of 2 h, the corresponding upflow velocity being 0.56 m/h. Such a situation was not encountered in the modified UASB reactor at this HRT. When the modified reactor was operated at 1.5 h HRT, the sludge bed was completely fluidised and the sludge even occupied the tube settlers, since no sludge was being wasted regularly from port No. 2 as was done in this investigation. Even at this situation when the expanded bed was occupying the settler zone, sludge washout did not occur (Gawande, 1996). These observations also indicated the improved capability of the modified configuration in retaining the suspended biomass in the reactor. The excellent effluent quality that was achieved at the

extremely low HRTs (1-3 h) with domestic wastewater (Table 6.5) calls for installation of pilot-plant for scale-up.

Table 6.5: Summary of the Reactor Performance for the Three Types of Wastewaters

| Parameter | Sucrose-based wastewater | | CERELAC-based wastewater | | | Raw domestic wastewater | |
|---|---|---|---|---|---|---|---|
| | $R_1$ | $R_2$ | $R_1$ | $R_2$ | | $R_1$ | $R_2$ |
| HRT (h) | 2-5 | 2-5 | 2-4 | 1 | 2-4 | 1-3 | 2 |
| Temperature ($^{o}$C) | 29-35 | 29-35 | 15-23 | 26 | 15-23 | 30-32 | 32 |
| Influent total COD (mg/L) | 485 | 485 | 476 | 476 | | 125-622 | |
| **Reduction** | | | | | | | |
| Total COD (%) | 84-90 | 83-90 | 81-83 | 47 | 81-82 | 79-87 | 78 |
| Total BOD (%) | 93-96 | 92-96 | 90-91 | - | 89-90 | 81-88 | 81 |
| Total TSS (%) | - | - | - | - | - | 94-95 | 95 |
| Closeness to ideal system in total COD removal (%) | 92-95 | 91-95 | 90 | 71 | 89 | 90-96 | 90 |
| **Effluent Characteristics** | | | | | | | |
| Total COD (mg/L) | 46-78 | 50-82 | 83-88 | 252 | 86-88 | 52-57 | 59 |
| Total BOD (mg/L) | 17-32 | 18-35 | 28-32 | - | 32-35 | 14-15 | 15 |
| TSS (mg/L) | 45-80 | 49-80 | 35-47 | 201 | 41-43 | 24-60 | 25 |

# 7. CONCLUSIONS

With the aim of minimising the loss of suspended solids with the effluent, a reactor configuration was conceived and designed by incorporating tube settlers in the settler zone of a UASB reactor replacing the conventional GLSS. Two bench-scale units of the modified configuration were fabricated in which an assembly of PVC tubes having 2 cm diameter and 54 cm length was provided above the digestion zone. Such an arrangement of tubes was expected to effect proper gas-liquid-solid separation resulting in effluents of low COD. Moreover, the configuration was expected to achieve loading rates conducive for granulation with low-strength wastewater without net loss of biomass during start-up. The two reactors $R_1$ and $R_2$ with the settlers inclined at angle of $45^o$ and $60^o$ respectively were used to develop granular sludge from a flocculant seed sludge with low-strength sucrose-based synthetic wastewater. The reactors containing the granular sludge were used to treat sucrose as well as CERELAC-based low-strength wastewater at HRTs ranging from 1-5 h. Further, the reactors were employed for treatment of raw domestic sewage of low-strength. Based on the above experimental investigations, the following conclusions may be drawn:

(1) Sedimentation of organic solids after the release of attached gas bubbles and its return to the digestion zone was effected by the tube settlers.

(2) Granulation was observed in the reactor while operating at 4 h HRT with sucrose-based wastewater. The space and sludge loading rates were 2.88 g COD/L.d and 0.24 g COD/g VSS.d respectively. The gas production rate in the reactors was about 0.9 L/L.d.

(3) With sucrose-based wastewater, the total and soluble COD removal efficiencies of 83-90% and 91-95% respectively were observed in the reactors at HRTs ranging from 2-5 h. The effluent total $BOD_5$ was in the range of 17-35 mg/L.

(4) The tube settlers were more effective in the removal of suspended solids from the effluent. However, the reduction in soluble COD was only marginal.

(5) During the reactor operation at 2-5 h HRT with sucrose-based wastewater, the average methane yield was 0.7 g $CH_4$-COD/g COD removed. The excess sludge production was 0.16 g VSS/g COD removed.

(6) The granular sludge developed on sucrose-based wastewater could be easily adapted to the CERELAC-based wastewater. After 45 days of secondary start-up the total and soluble COD removal efficiencies were more than 80%. The reactor pH was maintained above 7.0 without the addition of bicarbonate.

(7) During steady state operation at 4 h and 2 h HRT, the total COD removal efficiency was in the range of 81-83%. The total $BOD_5$ of the effluent was in the range of 28-35 mg/L.

(8) At 1 h HRT, the settler was not effective in achieving proper gas-liquid-solid separation.

(9) With the CERELAC-based wastewater, the methane yield was 0.47 g $CH_4$-COD/g COD removed and the sludge yield was 0.16 g VSS/g COD removed. The relatively low methane yield was attributed to the low BSRT in the reactor and the precipitated or adsorbed solids or fat.

(10) The granular sludge grown on CERELAC-based wastewater could be adapted easily to the domestic wastewater. Within 23 days of reactor operation at 3 h HRT, the maximum total and soluble COD removal efficiency that could be achieved in $R_1$ was 86% and 74%. In the case of $R_2$ which was operating at 2 h HRT, these efficiencies were 84% and 66%.

(11) When the reactors were fed with domestic wastewater, in spite of the wide fluctuation in the influent characteristics, the effluent characteristics were fairly stable.

(12) At HRTs ranging from 1-3 h, the average total COD removal was in the range of 78-87%. At 1 h HRT, the space loading rate was 9.65 g COD/L.d and the treatment efficiency achieved was 87%. The average total $BOD_5$ of the effluent was about 15 mg/L over the range of HRTs studied.

(13) The average effluent TSS at the range of HRTs studied varied from 24-60 mg/L. When the reactors were operated at 1 and 2 h without tubes, the average effluent TSS were 111 and 114 mg/L. This indicates the effectiveness of the tube settlers.

(14) No gas could be recovered during domestic wastewater feeding. This was attributed to the high insoluble fraction of the influent

COD, impaired stabilisation of suspended organics at the extremely low HRTs, and the methane lost with the effluent.

(15) The average excess VSS production observed was 0.14 g VSS COD/g $COD_{in}$ and the excess TSS production was 7.6 X $10^{-2}$ g TSS/g $TSS_{in}$ over the range of HRTs studied.

(16) After about 125 days of reactor operation with domestic wastewater, about 28% of the sludge solids were having a settling velocity (54 m/h), comparable to that of granular sludge.

(17) When the reactors were fed with the sucrose or CERELAC-based wastewater the SMA of the reactor sludge varied from 1-1.6 g $CH_4$-COD/g VSS.d. During domestic wastewater feeding, the SMA varied from 0.12-0.2 g $CH_4$-COD/g VSS.d. The reactor sludge VSS and VSS/TSS ratio were dependent on the type of wastewater.

(18) With all the three types of wastewater the modified UASB reactors were 90-96% close to the ideal system. When the reactor was operated at 1 h HRT without tubes, this closeness was only 46%.

(19) Apart from the flow through velocity the gas flow rate is also to be considered in the design of tube settlers.

(20) The high effluent quality obtained during this investigation suggests that this type of configuration is useful for treatment of domestic wastewater.

# 7. SUGGESTIONS FOR FUTURE WORK

Based on this investigation, the following suggestions may be made for future study:

(1) Experimental studies may be conducted to achieve granulation with medium strength domestic wastewater using the modified UASB reactor.

(2) Detailed studies may be conducted to delineate the role of gas production rate on granulation with low strength wastewaters of varying methane potential.

(3) Studies on the microstructure of granules at various stages of its development and subsequent aging is expected to give more insight to the phenomenon of granulation.

(4) There is a need to understand the inter-relationship between the diameter of tube, flow through velocity and gas loading rate. Experimental studies may be conducted to explore the above with various combinations of these three variables.

(5) Pilot-plant studies may be conducted to obtain various parameters for scale-up.

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